The evaluation of microstructure, grain boundary character and micro texture of [Al/Si₃N₄/Al₂O₃] P nanocomposites fabricated through PM route and its influence on compressive and three-body wear properties

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Abstract

The compressive properties and 3 body wear characteristics of powder metallurgical (PM) processed [Al/Si₃N₄/Al₂O₃] P Nanocomposites with single and combined reinforcement of Al₂O₃ and Si₃N₄ reinforcing particles having different compositions (1%, 2% and 3%) were studied and evolution of microstructure, grain boundary character and micro texture of fabricated [Al/Si₃N₄/Al₂O₃] P Nanocomposites was investigated through EBSD in the present research work. The fraction of high angle boundaries (HAGBs) were observed more in combined reinforcing samples of Al₂O₃ and Si₃N₄ whereas a single reinforcing sample of Al₂O₃ and Si₃N₄ showed fewer HAGBs. Micro texture results showed the strong textures components near to {112} 〈111〉 Cu and {110} 〈111〉 for pure sintered Al sample P and mixed reinforcement composites (M₁, M₂ and M₃) > P whereas for single reinforcing sample showed weak textures near to transverse direction. Out of all fabricated composites, 2% mixed Al₂O₃ and Si₃N₄ reinforced composite revealed the maximum ultimate compressive strength (209.98 MPa) and least wear rate (0.1 mm³/min mm²/N-cm for 1 kg load and 3.5 mm³/N-cm for 2 kg load) attributing formation of nanocluster causing grain boundary pinning effect. The dominant failure mechanism for all samples was also detected and found to be a mixed-mode ductile failure mechanism for 2% mixed Al₂O₃ and Si₃N₄ reinforcement composite while other sample failed through ductile as well as mixed-mode mechanisms.

1. Introduction

Aluminium-based metal matrix composite (AMMC) extensively used in automobile parts, aircraft structure and electronic devices due to their enhanced properties such as high specific strength, hardness, fatigue and fracture toughness compared to monolithic materials[1]. Out of all AMMCs, metal matrix composites based on nanoparticle reinforcement has emerged as a promising material and found most attention owing to the presence of hard and brittle nano reinforcement which provides better tribological properties, creep characteristics and cyclic fatigue/fracture response[2]. The combined advantage of matrix material and reinforcing particle may also offer the synergistic enhancement in material properties which is unachievable in unreinforced material.

Various techniques have been proposed to fabricate nanocomposites with improved mechanical and tribological properties and can be categorised into two ways: liquid state processing and solid-state processing. The liquid state processing techniques involve; squeeze/stir casting, pressure filtration, selective laser melting and ultrasonic-assisted casting[3, 4]. These processes typically involve the integration of nano reinforcing...
particles with molten matrix material and their subsequent solidification. However, the difficulty associated with the liquid state processing process could lead to a non-homogeneous distribution of nano reinforcement in the matrix and imperfect agglomeration which further causes brittleness in fabricated nanocomposite [5]. This limits the use of liquid state processing for the fabrication of nanocomposite. Solid-state processing methods such as spark plasma sintering, hot press sintering, hot isostatic pressing and powder metallurgy (PM) may be used as an alternative approach for the fabrication of nanocomposites due to their high working temperature and long processing time. In addition melting phenomenon is also avoided in such type of processes. Among all-solid-state processing methods PM has a great potential to develop nanocomposites and gained significant attention because of fabrication ease, microstructure and mechanical properties [6].

Recently reddy et al reported the mechanical properties of nano-sized SiC reinforced composite prepared by microwave sintering and found a significant increase in compressive (25%) and tensile properties (13%) of the fabricated composite after the addition of SiC nano reinforcement [7]. Dhanashekhar et al studied the mechanical and wear characteristics of AA6061/SiC composite manufactured through PM technique and reported the enhanced compressive and hardness values with an increasing percentage of reinforcing particles. Homogeneous distribution of reinforcing particle in the matrix was observed to be the primary reason for the improved mechanical properties as reported in their work [8]. Bongale et al fabricated the nanocomposite with varying composition of Al$_2$O$_3$ and SiC reinforcing particles by PM technique and observed the enriched mechanical and tribological properties after inclusion of SiC particle [9]. Kumar et al investigated the wear behaviour of Al/Al$_2$O$_3$ /SiC nanocomposite developed by PM method and stated that variation in % composition of reinforcing particles greatly influences the mechanical and wear behaviour of the fabricated composite. The additions of Al$_2$O$_3$/SiC up to 7% is the optimal composition for improved mechanical and wear characteristics in their study [10].

Among the different particle reinforcement, ceramic nanoparticle especially Al$_2$O$_3$ and Si$_3$N$_4$ have found to be an excellent reinforcing particle due to their high wear resistance, stiffness, high melting temperature, low density and high specific strength. In past research, the effort has been focused on refining the grain size of reinforcement into the nanoscale to achieve the superior mechanical properties caused by interaction between dislocation and nanoparticles [11, 12]. However, the discussions on the fabrication of nanocomposites and strengthening through nanoparticle reinforcement are still vague in the literature. In addition, it is well established that crystallographic orientation/texture greatly influences the mechanical properties and formability in Al alloy and Al-based nanocomposites. In general two types of texture studies are associated with Al alloys: (1) Deformation texture which typically associated with brass component \{110\} \langle 112 \rangle and Cu component \{112\} \langle 111 \rangle; (2) Recrystallization texture which is dominated by goss and cube components having \{110\} \langle 001 \rangle and \{001\} \langle 100 \rangle orientations respectively. Texture developments in Al alloys and Al-based metal matrix composites fabricated by SPD approach and other techniques are thoroughly studied however texture development in Al-based nanocomposites fabricated through PM less understood and scarce in the literature. According to the property moderation, the fabricated nanocomposite has industrial application in manufacturing of automotive structural parts like break and clutch linings. Despite their several benefits, there are various drawbacks in terms of industrial applications such as high manufacturing cost, weak interfacial bonding between reinforcement and matrix, inhomogeneous distribution of reinforcing particles and grain growth/coarsening.

In view of the above-mentioned discussions, it may be mentioned that most of the published research on Al-MMC with nanoparticle reinforcement is based on the tensile and tribological characteristics only, however, combined and detailed study of compressive properties, 3 body wear behaviour of Al-MMC and texture development with varying percentage of nano reinforcement of Al$_2$O$_3$ and Si$_3$N$_4$ fabricated by PM technique is limited in the literature. Therefore present work examines the impact of Al$_2$O$_3$ and Si$_3$N$_4$ nano particle reinforcement on the compressive properties, abrasive wear characteristics and texture development.

2. Material and methods

In this research work, very fine laboratory-grade aluminium powder of 600 meshes is used for matrix material with average particle size (APS) up to 20 microns. The reinforcing materials to fabricate the nanocomposites are nanoscale particles of silicon nitride (Si$_3$N$_4$) and alumina (Al$_2$O$_3$). Both as received nanoparticles are having a purity of 99.9%. To develop the nanocomposites, the powder metallurgy (PM) route was used under the compaction pressure of 12.06 MPa. The matrix and reinforced particles were premixed to develop green compacts according to the various wt% (1, 2 and 3 wt %) as shown and abbreviated in table 1.

The microstructural analysis of developed nanocomposites was done using Transmission Electron Microscopy (TEM) and Electron Backscatter Diffraction (EBSD). TEM investigations are carried out through
Philips CM 12 transmission electron microscope operating at 120 V. TEM samples for pure and fabricated composites were made by mechanical polishing and grinding the sample to 100 μm with the help of emery papers and polishing machine followed by thinning the sample by twin jet electro polishing with a solution of 20% sulphuric acid and 80% methanol at a temperature of −20°. Microstructural characterization using EBSD was carried out with the help of FEI QUANTA 400 FEG SEM. The samples were mechanically ground and polished through diamond paste with a fine sized colloidal silica suspension for sufficient time. EBSD data measurement using an orientation map was performed by taking a minimum step size of .03. The TSL OIM software was used for EBSD analysis. Grain size measurement and orientation of grains were characterized through an inverse pole figure (IPF) map. Texture information and grain size are related to the different colour associated with the IPF map. For the identification of grain boundary misorientation angle, a boundary misorientation histogram was also plotted. If the adjacent grains have a misorientation angle (θ) of 2° ≤ (θ) ≤ 10° then those grains were considered as low angle grain boundaries (LAGBs) whereas the misorientation angle (θ) ≥ 10° was treated as high angle grain boundaries (HAGBs).

The phase identification of all fabricated nanocomposites was done using x-Ray diffraction (XRD) subjected to the diffraction angle 2θ (20° to 80°). To analyse the monotonic behaviour of prepared nanocomposite samples, the compressive test was carried out for all sample conditions. The test samples were prepared as per ASTM standard E9 [13]. To analyse the tribological behaviour of the fabricated nanocomposite sample, three body wear test was performed on each sample under varying loading condition (1 kg and 2 kg) for a constant time of 10 min. To confirm the mechanism of wear the scanning electron microscopy (SEM) was done post experimentation.

Table 1. Compositions & abbreviations of various nanocomposite samples.

| S. No | Abbreviations | Constituents | Reinforcement Wt % |
|-------|---------------|--------------|-------------------|
| 1     | P             | Al Pure      | 100               |
| 2     | A1            | Al with Al₂O₃| 1                 |
| 3     | A2            | Al with Si₃N₄| 2                 |
| 4     | A3            | Al with Si₃N₄&Al₂O₃| 3             |
| 5     | S1            | Al with Si₃N₄| 1                 |
| 6     | S2            | Si₃N₄         | 2                 |
| 7     | S3            | Si₃N₄         | 3                 |
| 8     | M1            | Al with Si₃N₄&Al₂O₃| 1             |
| 9     | M2            | Si₃N₄         | 2                 |
| 10    | M3            | Si₃N₄         | 3                 |

Figure 1. XRD graph for various compositions of fabricated nanocomposites.
3. Results

3.1. XRD characterization

Figure 1 shows the XRD graphs for all nanocomposites. The well-structured aluminium phase is observed by XRD characteristics in all fabricated samples as evidenced by sharp diffraction peaks. For 1 wt% Al2O3 (A1) reinforced sample condition very small size peaks are clearly visible which confirms the presence of A1. With an increasing percentage of Al2O3 reinforcement from 1 wt% to 3 wt% the gradual rise in peak intensities were observed (figure 1). Similarly very small size peak of sample condition S1, i.e. 1 wt% reinforcement of Si3N4 was noticed in XRD graph (figure 1). As the percentage reinforcement of Si3N4 increases from 1 wt% to 2 and 3 wt%, peak intensity increases correspondingly as depicted from figure 1. Moreover for the sample conditions M1, M2 and M3 both Al2O3 and Si3N4 phases were observed, however with increasing percentage of these phases, gradual rise in the peak intensity is noticed from M1 to M3 sample conditions.

3.2. Microstructural evolution

3.2.1. Transmission electron microscopy (TEM)

TEM micrographs for PM processed fabricated composites samples are shown in figure 2. TEM microstructure for A1, A2 and A3 sample composition is shown in figures 2(a)–(c). Existence of spherical shape Al2O3 nano reinforcement (size less than 100 nm) can obviously be envisaged in these micrographs. Apart to this, these particles are thoroughly and homogeneously dispersed in Al matrix as noticed from TEM microstructure. However volume fraction of these nano size reinforcement increases gradually with increase in wt% from 1 to 3 as observed from TEM Figures (figures 2(a)–(c)). It is believed that compacting and sintering during PM process has led to the uniform dispersion of particles and having significant role in property moderation in fabricated composite. Representative TEM micrographs for S1, S2 and S3 sample composition are displayed in figures 2(d)–(f). The nanometric inclusions of Si3N4 can be clearly visualized in these Figures. However the dispersion of these particles with the Al matrix is not observed to be uniform and homogeneous as in case of samples A1, A2 and A3. TEM micrographs for M1, M2 and M3 sample composition are presented in figures 2 (g)–(i). Al2O3 and Si3N4 nanometric reinforcement can be envisaged in each of illustrative TEM
micrographs i.e. Figures 2 (g)–(i). Some sign of clustering of these nanoparticles are also observed in these figures. M₂ sample composition has shown uniform clustering of the particles in the present work as seen in TEM micrograph. Apart to this fine ceramic nanometric inclusions in all samples of M₁, M₂ and M₃ are homogeneously dispersed and preferably located in grain boundaries as noticed from TEM pictures of respective samples. However grain boundary pinning effect is observed to be more in M₂ sample composition in this work.

3.2.2. Electron back scattered diffraction (EBSD)

Microstructure characterisation for pure and fabricated nanocomposites were performed by using Electron back scattered diffraction (EBSD) technique in the present work. The microstructural details and grain size distribution of pure sintered Al sample (P) and fabricated nano composites as achieved from EBSD analysis, are displayed in figures (3–12). Figures 3(a)–(d) shows the EBSD results of pure sintered Al (P) in the form of inverse pole figure (IPF) map, image quality (IQ) map, grain size distribution chart and boundary misorientation histogram. The microstructure of pure sintered Al (P) sample consisted of variety of grain sizes ranging from 10 μm to 40 μm having average grain size of 33 μm as observed from figure 3(c). The histogram of boundary misorientation angle in figure 3(d) represents and reveals the random microstructure having low fraction of low angle boundaries (LAGBs) and more fractions of high angle boundaries for pure sintered Al sample. Low fraction of low angle grain boundaries usually associated with lower deformation of metals/alloys as ascribed in various literatures [14] which confirms that pure Al sample persisting of coarser grains.

Figures 4 (a)–(d) shows the EBSD results of 1% Al₂O₃ (A₁) sample composition. As noticed in figures 4 (a)–(c), the microstructure of 1% Al₂O₃ (A₁) sample consisted of slightly uniform microstructure having grain size ranging from 100 nm to 150 nm with an average grain size of 127 nm. Inverse pole figure (IPF) and image quality (IQ) map corresponding to this sample composition shows the large number of black points in the figure 4(a). These black point in the figures may be the unindexed point due to porosity of the sample. Grain size distribution chart for A₁ sample reveals that more than 76% grain have a grain size lesser than 1 μm. Boundary misorientation histogram in figure 4(d) represents the high fraction of low angle grain boundary (LAGBs) while lower fraction of high angle grain boundaries (HAGBs) which validates the presence of nanometric microstructure in this sample composition.

Figure 3. Pure sintered aluminium sample (a) IPF map (b) IQ map (c) grain size distribution chart (d) Boundary misorientation histogram.
EBSD results for 1 wt% Si₃N₄ i.e. S₁ sample composition are shown in figures 5(a)–(d). IPF and IQ maps corresponding to this sample condition in figures 5(a)–(b) clearly reveals the nanometric microstructure having grain size in the range of 100 nm to 200 nm with an average grain size of 135 nm. Grain size distribution chart for S₁ sample composition is presented in figure 5(c) which clearly confirms that large fraction of grains in sample are sizes lesser than 1 μm. Boundary orientation histogram as seen in figure 5(d) for this sample composition represents the large fraction of LAGBs and slight increase in fraction of HAGBs with respect to A₁ sample composition which indicates the better refinement and homogeneity in the Si₃N₄ reinforced composite.

EBSD results for 1% mixed Al₂O₃ and Si₃N₄ i.e. M₁ sample composition are presented in figures 6(a)–(d). As observed in figures 6(a)–(b), which are IPF and IQ maps of M₁ sample, the significant changes in the microstructure was noticed here. The microstructure consisted of wide range of grain size ranging from 100 nm to 12 μm with an average grain size if 780 nm. In addition, the random orientation of grains are also observed for M₁ sample composition as noticed from IPF and IQ maps. This might be due to inhomogeneity during sintering and mixing process of two indifferent reinforcing particles in the present work. IPF and IQ maps also reveals that number of fractions of grains which are sizes lesser than 1 μm are lower as compared to 1% Al₂O₃ reinforcement (A₁). The reduced fraction of black spot or unindexed spots also confirms that with increasing wt % of Al₂O₃ particles porosity level in sample decreases gradually. Grain size distribution chart for A₂ sample composition is presented in figure 7(c). In this case microstructure consisted of grain sizes ranging from 100 nm to 140 nm with an average grain size of 119 nm as predicted from grain size distribution chart (figure 7(c)). In addition, from IPF, IQ and grain size distribution chart, it is also noticed that more than 85% grain attains the grain size below than 1 μm which clearly indicates the more homogeneous and finer microstructure for A₂ sample. Boundary misorientation histogram for A₂ sample composition is shown in figure 7(d). High fraction of LAGBs and lower
Figure 5. 1 wt% reinforcement of Si$_3$N$_4$ sample (a) IPF map (b) IQ map (c) Grain size distribution chart (d) Boundary misorientation histogram.

Figure 6. 1 wt% mixed reinforcement of Al$_2$O$_3$ and Si$_3$N$_4$ sample (a) IPF map (b) IQ map (c) Grain size distribution chart (d) Boundary misorientation histogram.
fraction of HAGBs can clearly be evidenced from this figure which designates the presence of nanometric microstructure and higher deformation strain in fabricated composite.

Figures 8(a)–(d) shows the EBSD results of 2% Si₃N₄ (S₂) sample composition. As seen in figures 8(a)–(c), IPF map, IQ map and grain size distribution chart clearly indicates that microstructure consisted of wide variety of grain size ranging from 100 nm to 300 nm with an average grain size of 147 nm. Boundary misorientation histogram from figure 8(d) also represent slight decrease in fraction of LAGBs while considerable increase in fraction of HAGB.

EBSD results for mixed 2% Al₂O₃ and Si₃N₄ (M₂) sample composition are shown in figures 9(a)–(d). As seen in figures 9(a)–(b) for IPF and IQ map, microstructure consisted of various ranges of grain size and catagorised in three different zone. One zone consisted of very fine nanometer level grain structure having grain size in range of 40 to 100 nm. In another zone grain size lies in the range of 100 nm to 700 nm, while in third zone more than 700 nm to 5 μm grain sizes are observed. IPF and IQ map also gives the glimpse of recrystallization of sample in this case. Grain size distribution chart for this sample composition is shown in figure 9(c) which clearly indicates the wide grain size distribution for this condition. The quantitative data for the fraction of high and low angle grain boundaries for this sample composition is represented by boundary misorientation histogram in figure 9(d). The fraction of LAGBs slightly decreases and fraction of HAGBs has noticeably been increased as observed in figure which clearly validates that recrystallization process has occurred in this sample composition.

EBSD results for sample A₃, S₃ and M₃ are shown in figures 10–12. On close observing these results it can be mentioned that on single reinforcement of Al₂O₃ and Si₃N₄ up to 3%, microstructure remains in homogeneous state and on hybrid reinforcement of Al₂O₃ and Si₃N₄ up to 3% wide range of grain sizes were observed as seen in figures (10–12). The average grain size for A₃, S₃ and M₃ were noticed as 167 nm, 225 nm and 810 nm. The boundary misorientation histogram for A₃ and S₃ condition represents high fraction of LAGBs while low fraction of HAGBs, on the other hand M₃ sample composition shows just reverse of it i.e. low fraction of LAGBs and high fraction of HAGBs.

3.2.3. Texture development

{001}, {110} and {111} pole figures (PFs) for pure sinterd Al sample (P) are shown in figure 13(a). These PFs shows fully recrystallized microstructure with texture near to transverse direction (TD). High fraction of HAGBs
Figure 8. 2 wt% reinforcement of Si₃N₄ sample (a) IPF map (b) IQ map (c) Grain size distribution chart (d) Boundary misorientation histogram.

Figure 9. 2 wt% mixed reinforcement of Al₂O₃ and Si₃N₄ sample (a) IPF map (b) IQ map (c) grain size distribution chart (d) boundary misorientation histogram.
Figure 10. 3 wt% reinforcement of Al₂O₃ sample (a) IPF map (b) IQ map (c) Grain size distribution chart (d) Boundary misorientation histogram.

Figure 11. 3 wt% reinforcement of Si₃N₄ sample (a) IPF map (b) IQ map (c) Grain size distribution chart (d) Boundary misorientation histogram.
Figure 12. 3 wt% mixed reinforcement of Al₂O₃ and Si₃N₄ sample (a) IPF map (b) IQ map (c) grain size distribution chart (d) boundary misorientation histogram.

Figure 13. 001, 111, and 110 pole figures for (a) pure sintered sample (b) 1 wt% reinforcement of Al₂O₃.
from IPF map corresponding to this sample condition (figure 3(d)) also validates the presence of recrystallised texture in this case [15–17]. In addition maximum intensity of recrystallized texture is 9.4 as seen from figure 13(a). These textures also represents that preferred texture componants in the sample are near to \{112\} \langle111\rangle Cu and \{110\} \langle111\rangle P for pure sintered Al(P) sample. It is believed that copper componant may be produced during premixing and compacting process while phosphorous componant is evolved during sintering process.

Pole figures (PFs) for A1, S1 and M1 sample compositions are shown in Figure 13 (b) and figures 14(a), (b). PFs for single 1% reinforcement of Al2O3 and Si3N4 samples i.e. A1 and S1 shows unrecrystallised microstructure with texture near to transverse direction (TD). The presence of high fraction of LAGBs as seen in EBSD IPF map also confirms the same. In addition intensity of preferred texture component for both samples A1 and S1 significantly reduced to 1.5 as noticed from figure 13(b) and figure 14 (a). The drastic drop in texture intensity supports the claim of random orientation of crystallographic planes in the present work [15–17]. The significant change in the texture development is observed for the 1% mixed reinforcement of Al2O3 and Si3N4 (M1) sample composition as seen in figure 14 (b). The PFs for this sample composition shows recrystallised microstructure with strong texture near to transverse direction (TD). The observed preferred texture component in the sample are near to \{112\} \langle111\rangle Cu and \{110\} \langle111\rangle P as in case of pure sintered Al(P) sample. On comparing the texture intensity with respect A1 and S1 samples, it is seen that intensity reaches from 1.5 to 2.7 for M1 composition. The improvement in texture intensity for M1 composition clearly explains that mixed reinforcement has certainly preferred orientation in TD. Moreover high fraction of HAGBs as observed in EBSD IPF map validate the trace of recrystallization during PM processing of this sample.

{001}, \{110\} and \{111\} pole figures (PFs) for A2, S2 and M2 sample compositions are represented in figures 15(a), (b) and 18 (a) respectively. PFs for A2 sample composition having 2% Al2O3 are shown in figure 15 (a). As seen in figure 15 (a), A2 sample has a weak texture in TD as in case of A1 sample (figure 13 (b)). EBSD IPF map for this sample also does not show any kind of dynamic recrystallisation process as observed in figure 7 (a). The complete microstructure pertains nano sized grains with large number of LAGBs as noticed from grain size distribution chart (figure 7 (c)). It may be mentioned that with increasing % reinforcement of Al2O3 recrystallisation process could not occur resulting weakening of texture. In the present work, texture intensity level for A2 sample was observed as 1.5 same as in case of A1 sample. Similarly PFs for S2 sample with 2% Si3N4 addition are dislpayed in figure 15 (b). It is noted that with increasing % of Si3N4 dynamic recrystallisation is believed to occur as noted in \{001\}, \{110\} and \{111\} pole figures and EBSD IPF map (figures 8 (a)). High fraction
of HAGBs in this case also reported the same as seen from grain size distribution chart (figure 8 (c)). On comparing the texture intensities with single reinforcement of Al₂O₃ and Si₃N₄ (A₁, S₁), it is found that texture intensity rises from 1.5 to 1.9 which indicates that S₂ sample has slight preferred orientation near to {112} (111) Cu and {110} (111) P. PFs for M₂ sample with 2% mixed compositions of Al₂O₃ and Si₃N₄ are shown in figure 16 (a). PFs of M₂ sample clearly reveals the substantial change in preferred orientation and texture development. It is also noted that dynamic recrystallization process has been accelerated with % increase of mixed reinforcement of Al₂O₃ and Si₃N₄ up to 2% as observed in figure 16 (a). More fraction of HAGBs (figure 9 (d)) corresponding to this sample composition also validates the same. On close revealing the texture intensity of M₂ sample, the texture intensity reaches to 3.7 indicating strong texture towards TD direction.

Similarly {001}, {110} and {111} pole figures (PFs) for A₃, S₃ and M₃ sample are shown in figure 16(b) and figures 17 (a)–(b) respectively. The observed trend for texture development for all these samples were noticed same as in case of A₁, S₁, M₁ and A₂, S₂, M₂ in the present work. In this case single reinforcement samples of 3% Al₂O₃ and Si₃N₄ has shown slightly improved texture intensities of 2.1 and 2.3 with respect to single reinforcement of 1% and 2% Al₂O₃ and Si₃N₄. However with 3% mixed Al₂O₃ and Si₃N₄ sample drastic increase in texture intensity 4.9 was observed which substantiate the strong texture of M₃ sample in TD as compared to rest of fabricated nanocomposite samples. It is also noticed that with increasing % of mixed reinforcement up to 3% recrystallisation mechanism has prominence effect on the microstructure as seen in IPF and grain size distribution chart (figures 12 (a)–(c)).

3.3. Mechanical behaviour

3.3.1. Hardness

Hardness is very useful property that provides the information about the strength and tribological behaviour of processed material. In general various factors such as particle shape/size, volume fraction, dispersal of reinforcing phase, grain boundary nature, texture involved and preparation method may affect the hardness of fabricated composite as reported in literature [8, 18]. Figure 18 represents the average hardness versus compositions graph for all fabricated nanocomposites. Hardness of pure sintered Al sample (P) has a value of 48 HV and found to have lowest hardness values with respect to rest of samples. The lower hardness of pure Al sample may be correlated with EBSD microstructure in section 3.2.2, where coarser grains with average grain size of 40 μm along with high fraction of HAGBs were seen. Coarser grain and high fraction of HAGBs might be the reason for lower hardness of pure sintered Al sample in the present work [19]. The sample A₁ in figure 18...
Figure 16. 001, 111, and 110 Pole figures for (a) 2 wt% mixed reinforcement of Al$_2$O$_3$ and Si$_3$N$_4$ (b) 3 wt% reinforcement of Al$_2$O$_3$.

Figure 17. 001, 111, and 110 Pole figures for (a) 3 wt% reinforcement of Si$_3$N$_4$ (b) 3 wt% mixed reinforcement of Al$_2$O$_3$ and Si$_3$N$_4$ sample.
shows the hardness of 53 HV which is 10.4% increment in hardness value as compared to pure sintered Al (P) sample. TEM microstructure (figure 2(a)) corresponding to this sample condition has shown fine spherical size nano reinforcement of Al₂O₃, which is discussed in detail in section 3.2.1. This nano size reinforcement may effectively pin the grain boundary and might become strong obstacle for dislocation motion as stated in various literatures causing higher hardness of the sample [19, 20]. In addition, nano sized grains having average grain size 127 nm and presence of more fraction of LAGBs as discussed in EBSD section 3.2.2 may also provide the additional obstruction to dislocation movement resulting higher hardness of A₁ sample. Hardness values for S₁ and M₁ samples are observed as 50 and 51 HV respectively and can be seen in figure 18. It is seen that there is not major difference in hardness of S₁ and M₁ samples, however the observed hardness values are (50 and 51 HV) lesser with respect to A₁ sample. The drop in hardness of S₁ and M₁ sample may be attributed to increase in fraction of HAGBs and slight coarsening of grains as observed and discussed in EBSD (figures (5), (6)) IPF, IQ and grain distribution histogram. The lower hardness values of S₁ and M₁ sample can also be correlated with TEM microstructure as described in section 3.2.1. It is reported that size and fraction of reinforcement, second phase particles greatly influence the mechanical properties. A fine reinforcement/second phase particle with large volume fraction imparts greater strengthening effect rather than coarser reinforcing particles. Similar phenomenon might be the reason for the low hardness of S₁ and M₁ sample as described in TEM microstructure corresponding to this sample composition. Figure 18 clearly reveals that 2 wt% mixed reinforced nanocomposite i.e. M₂ sample composition is having maximum hardness of 56 HV out of A₂, S₂ and M₂ sample composition whereas the A₂ and S₂ sample compositions shows the hardness of 53 and 50 HV respectively. High
hardness of M2 sample may be due to the Nano metric microstructure observed in EBSD IPF and IQ map as noticed in figures 9(a)–(c). Further TEM microstructure for M2 sample as seen in figure 2(h) also confirms and validates the higher hardness due to uniform clustering of nano reinforcement. It is reported that uniform clustering of nano reinforcement act as an obstacle for the dislocation motion and impose strengthening in the material [21] due to which M2 sample has shown higher hardness value in the present work. Similarly lower hardness values of A3 (50 HV), S3 (50 HV) and M3 (51 HV) can be explained and correlated with TEM and EBSD microstructure where more fraction of HAGBs, recrystallization effect and coarser reinforcing particles were observed.

3.3.2. Compressive properties and post fracture surface morphology of fabricated composite

Figure 19 shows the variation in load with respect to extension for compression test of all fabricated nanocomposite samples along with pure aluminium sample. The responses with respect to compositions provide the moderation in compression property. From figure 19 pure sintered samples have an ultimate compressive strength (UCS) of 81.82 MPa and failure strain 77.54%. This may be due to the coarser grain and high fraction of high angle grain boundaries which provide good properties under compressive loading.

Fractured SEM images of pure sintered Al sample is represented in figure 20(a). Post fracture surface morphology of pure sintered aluminium sample (P) clearly shows the large sized dimples ascribing ductile fracture behaviour under compressive loading resulting lesser compressive strength [22]. On addition of 1 wt% Al2O3 (A1) and 1 wt% Si3N4 (S1), UCS becomes 207.86 MPa and 183.94 MPa whereas failure strain reaches to 72.39% and 75.08% respectively. Post fracture surface morphologies of these samples are shown in figures 20(b)–(c). A1 and S1 sample fracture surface consists of finer ductile dimples, river pattern and brittle facets as evident from figures 20(b) and (c). However dimpled features are more to be observed in S1 sample. Such type of failure mode may be considered as mix mode failure as reported in various literatures [14, 22]. In addition dimpled like features in fractured surface indicates the material’s plasticity and can be interrelated with the observed fracture strain (75.08%) of the sample. Higher failure strain (72.39%) of A1 sample might be due to the ductile dimples in fractured surface which accumulate higher strain hardening effect during compressive testing with respect to S1 sample composition in this work. The UCS and corresponding failure strain of M1 sample is shown in figure 19. The M1 sample shows the UCS of 183 MPa while very less failure strain of 7.5% as evident.

Figure 20. Fracture surface (a) Pure Sintered aluminium (b) 1 wt% reinforcement of Al2O3 (c) 1 wt% reinforcement of Si3N4 (d) 1 wt% mixed reinforcement of Al2O3 and Si3N4.
from this figure. On comparing the post fractured surface of M1 sample with A1 and S1 sample, M1 sample’s fractures surface is covered with majority of brittle facets, river pattern and micro cracks as noticed from figure 20(d). Moreover fractured compressive surface of M1 sample reveal micro crack at an angle of 45° to the compressive test axis attributing the shear mode fracture in the present case. It is reported that shear mode fracture predominantly occurs due to the heterogeneous deformation and decreased rate of work hardening [23]. Similar behaviour might be the reason for the shear failure and observed lesser failure strain (7.5) of the M1 sample due to poor debonding of 1 wt% mixed Al2O3 and Si3N4 nano reinforcing particles with matrix.

Variation of UCS and failure strain for A2, S2 and M2 sample compositions are shown in figure 19. A2, S2 and M2 sample have the UCS values of 199.17 MPa, 145 MPa and 209.98 MPa respectively while their corresponding failure strain was noticed as 39.34, 34.98 and 69.77. Post fractured surface morphologies of A2 sample compositions as seen in figure 21(a) clearly depicts the presence of fine dimples along with brittle facets for A2 sample. However the fraction of dimpled covered region is more compared to brittle region substantiating mix mode ductile failure mechanism. On the other hand fractured surface of S2 sample perturbed with brittle facets and micro cracks at an angle of 45 degree to the test axis as evidenced from figure 21(b). Therefore shear mode fracture mechanism can be noticed for sample composition S2 in the present work. The observed failure strain for A2 and S2 validates this indifferent failure behaviour of these samples as seen from figures 21(a), (b). It may be mentioned that Al2O3 and Si3N4 particles fractured in the dimpled wall and brittle zone may provide the crack initiation cite causing mix mode and shear fracture mechanism for A2 and S2 sample composition respectively.

Figure 21(c) shows the fracture surface morphology for M2 sample composition after compression test. Fracture surface of M2 sample entirely covered with dimpled like features with little amount of brittle zone as seen in this figure. The work hardening ability of this sample composition is believed to be increased significantly with respect to A2 and S2 sample composition and can be confirmed from corresponding failure strain. In addition 2 wt% mixed Al2O3 and Si3N4 has shown better interfacial bonding, agglomeration and clustering with matrix in the present work which promotes void formation causing more fraction of dimpled features in fractured surface. It is also reported that excellent interfacial bonding between reinforcing particles and matrix improves the effective load transfer between them and encourages the work hardening behavior [24]. Similar phenomenon might be the reason for improved failure strain of M2 sample composition in this work.
UCS values and corresponding failure strains for A3, S3 and M3 samples are shown in figure 19. UCS values for A3, S3 and M3 samples are observed as 137.96 MPa, 138.96 and 195.82 MPa while their respective failure strain was noticed as 75.08, 18.14 and 72.39. For correlating compressive properties with the failure mode, the post fracture surface investigations was carried out through SEM. Figures 22 (a)–(c) shows the fractured surface morphology for sample composition A3, S3 and M3 respectively. Fractured surface for corresponding sample compositions obviously depicts the ductile failure mode in A3 and mixed mode failure in M3 sample composition while brittle failure mode in sample composition S3 as noticed from figures 22(a)–(c). Higher failure strain of A3 and M3 samples may be due to ductile and mixed mode failure behaviour of these samples which accumulate sufficient plasticity before the fracture of these samples.

3.4. Tribological behaviour (three body wear)

Tribological behaviours of fabricated nanocomposites were observed by three body wear test. In general, sand particles are used as hard protuberances which are forced against the surface of fabricated nanocomposites samples and produces abrasive wear in the sample. Figure 23 shows the specific wear rate with respect to all compositions of pure and fabricated nanocomposites (P to M3). The specific wear rates were studied with varying load (1kg and 2 kg) while other factors are kept constant (travelling distances 1.38 Km and sliding speed 2.3 m s⁻¹). Figure 23 clearly reveals that pure aluminium sample has shown the maximum wear under both loading conditions whereas the 2 wt% mixed reinforced nanocomposite (M2) reflects the minimum wear rate (0.1 mm³/min mm²/N·cm for 1 kg load and 3.5 mm³/N·cm for 2kg load) among all fabricated nanocomposite samples for both loading conditions.

3.4.1. Wear surface morphology

Worn surface morphologies of pure sintered Al sample (P) and all fabricated composites are shown in figures (24)–(26). The hardness of fabricated nanocomposites provides property moderation in the form of wear resistance as discussed in section 3.3.1. Hardness of the material is inversely proportional to the wear property as per the archard wear equation reported in the literature [25, 26] i.e., greater the hardness lower the wear and vice versa. It has been observed in the present research work that the wear rate is inversely proportional to the
hardness of nanocomposite. Worn surfaces of pure (P) and all 1 wt% reinforcement samples i.e. A₁, S₁ and M₁ are displayed in figure 24. It is depicted from figure 24(a) that pure aluminium sample have maximum wear rate due to lesser hardness and maximum ductility [26]. The wear surface morphology reveals the wear debris in the form of chips due to cutting action. The wear mechanism is totally plastic flow which results large size chips ploughed by harder asperity. In the category of 1 wt% reinforcement nanocomposites all three compositions (A₁, S₁ and M₁) shows brittle nature (figures 24(b)–(d)). Crack propagation has been clearly seen on the surface of all 1 wt% reinforcement nanocomposite samples as observed in figure. Sample S₁ have shown maximum wear.
rate whereas A1 sample is having least wear rate among all 1% reinforcement composites as seen in figure 23. This can be confirmed through respective worn surfaces of A1, S1 and M1 samples in figures 24(b)–(d) where higher surface damage effects along with pull off of reinforcing particles and majority of cracks were noticed for S1 sample composition. EDX images has also been shown as an inset for pure and all 1% reinforcement composites in figures 24(b)–(d). From EDX of all 1% reinforcement samples (A1, S1 and M1), it is clearly observed that crack nucleation begins at the respective reinforcing particles [27]. It is believed that when the hard asperity slides against the nanocomposite sample then the brittle nanocomposite could not accommodate the large amount of strain energy resulting development of sub surface crack [28]. Those sub-surface cracks propagate and reaches to the upper surface of material and hence material removal occurs.

In case of 2 wt% reinforced nanocomposites (A2, S2 and M2) sample composition, the sample M2 has shown least wear rate among all 2 wt% reinforced sample conditions, however S2 sample composition is having maximum wear rate as displayed in figure 23. The observed wear behaviour of 2 wt% reinforced samples can be correlated with worn surface morphologies of respective samples in figures 25(a)–(c) Worn surface of sample composition A2 clearly depicts the wear debris and micro crack in figure 25(a). In addition, particle pull off can clearly be evidenced from the worn surface which can be validated through EDX image shown as an inset in figure 25(a). Similar features were also noticed for sample composition S2 along with large size chips as depicted in worn surface of sample S2 in figure 25(b). However the surface damage effects were observed more in sample composition S2 as compare to A2 Sample composition. Worn surface of M2 sample composition is shown in figure 25(c). Worn surface of M2 sample composition have very few wear debris and lesser surface intrusions. Moreover wear particle plugging takes place through plastic flow wear mechanism as observed in figure 25(c). In the present work it may be mentioned that A2 and S2 is having mixed mode wear mechanism whereas M2 reflects ductile nature resulting plastic flow wear mechanism. Sample composition M2 reflects maximum hardness and have minimum wear rate as compared to all compositions of nanocomposite.

Figures 26(a)–(c) represents the worn surface of 3 wt% reinforced nanocomposite. Sample composition A3 and S3 shows mixed mode wear mechanism where crack growth, grain pull off and wear debris was observed. Sample composition M3 reflects brittle nature and hence only large number of sub cracks and micro crack was visible on worn surface.
4. Conclusion

Out of all fabricated composites sample 2 wt% mixed Al$_2$O$_3$ and Si$_3$N$_4$ exhibited maximum UCS (209.98 MPa) and hardness (56.6 HV) values as compared to the other samples. TEM microstructure for 2 wt% mixed Al$_2$O$_3$ and Si$_3$N$_4$ reinforced sample showed nanometric microstructure with uniform clustering of mixed reinforcement (i.e. Al$_2$O$_3$ and Si$_3$N$_4$) while for other samples clustering was not dominant. EBSD results showed the high fraction of HAGBs and low fraction of LAGBs for pure sintered Al sample P and mixed reinforcement composites (M1, M2 and M3) while for rest samples fraction of HAGBs and LAGBs were just vice versa. Texture results for pure sintered Al sample P and mixed reinforcement composites (M1, M2 and M3) showed the strong textures components near to $\{110\}$ $\langle 111 \rangle$ Cu and $\{110\}$ $\langle 111 \rangle$ P whereas for other conditions weak textures were observed. Fracture surface morphology of 2 wt% mixed Al$_2$O$_3$ and Si$_3$N$_4$ sample clearly exhibited the mixed modeductile failure mechanism whereas that the surface is entirely covered with dimpled like features with little amount of brittle zone with increasing work hardening ability. Wear rate under under three body wear conditions for 2 wt% mixed Al$_2$O$_3$ and Si$_3$N$_4$ sample composition was found to be least (0.1 mm$^3$/min mm$^3$/N-cm for 1 kg load and 3.5 mm$^3$/N-cm for 2kg load) out of all other sample compositions. Wear surface morphology of 2 wt% mixed Al$_2$O$_3$ and Si$_3$N$_4$ sample condition revealed the few wear debris and lesser surface intrusions in worn surface causing wear particle plugging and plastic flow wear mechanism.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).
**Declarations**

- Ethics approval and consent to participate
  
  Not Applicable

- Consent for publication:
  Not Applicable

- Availability of data and materials

  The authors confirm that the data supporting the findings of this study are available within the article [and/or] its supplementary materials.

**Competing interests**

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**Authors’ contributions**

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**Compliance with ethical standards**

**Conflict of interest**

There is no conflict of interest.

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