We are IntechOpen, the world’s leading publisher of Open Access books
Built by scientists, for scientists

4,100
Open access books available

116,000
International authors and editors

125M
Downloads

154
Countries delivered to

TOP 1%
Our authors are among the most cited scientists

12.2%
Contributors from top 500 universities

WEB OF SCIENCE™
Selection of our books indexed in the Book Citation Index in Web of Science™ Core Collection (BKCI)

Interested in publishing with us?
Contact book.department@intechopen.com

Numbers displayed above are based on latest data collected.
For more information visit www.intechopen.com
Chapter 1

Structural Characterization and Mechanical Behavior of Al 6061 Nanostructured Matrix Reinforced with TiO_2 Nanoparticles for Automotive Applications

S. Sivasankaran and Abdulaziz S. Alaboodi

Additional information is available at the end of the chapter

http://dx.doi.org/10.5772/65947

Abstract

The main aims of the present chapter are: learn synthesis procedure of AA 6061-x wt.% TiO_2 nanocomposites (x = 0, 2, 4, 6, 8, 10 and 12 wt.%) by mechanical alloying (MA); investigate structural characterization of manufactured nanocomposite powders using X-ray line profile analysis, scanning electron microscope (SEM) and transmission electron microscope (TEM); examine consolidation method and mechanical behavior in terms of sintered density, Vickers hardness and compressive stress-strain behavior; study the improvement of ductility in nanocomposites; and simulate the mechanical behavior using ANSYS. Here, the synthesized nanocomposites via MA were consolidated using conventional uniaxial die compaction; then, the green compacts were sintered at different temperatures. TEM microstructures of as-milled powder samples showed the matrix crystallite sizes ranging from 45 to 75 nm, which depended on the amount of reinforcement. A remarkable decrease in matrix powder particles size with the function of reinforcement was observed due to the ceramic nano TiO_2 particles acted as milling agent. The sintered nanocomposites yielded maximum strength of 1.126 GPa. The study of tri-modeled composite and its mechanical behavior revealed the possibility of achieving improvements in ductility and toughness for nanocomposites. The simulated mechanical behavior results using finite element method were good agreement with experimental results.

Keywords: mechanical alloying, nanocrystallite matrix, nanoparticles, characterization, mechanical behavior

© 2016 The Author(s). Licensee InTech. This chapter is distributed under the terms of the Creative Commons Attribution License (http://creativecommons.org/licenses/by/3.0), which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.
1. Introduction

Production of suitable materials to make our lives as comfortable, scientific development is playing a major role nowadays. Materials science and engineering is establishing the appropriate relationship between atomic structure and physical properties of materials which would design a novel material to attain improved properties [1]. The best performances of most of the components can be achieved in the present scenario by nanocomposites. The combined properties of metallic and ceramic materials compared with monolithic alloys can be achieved by metal matrix composites (MMCs) as it possesses high tensile strength, fatigue resistance, wear resistance and high elastic modulus. Several methods, namely, powder metallurgy (P/M), stir casting, pressure infiltration etc., have been developed for manufacturing of MMCs. In aerospace, space, automotive and structural parts, particulate lightweight aluminum-based nanocrystallite matrix reinforced with nanoparticles have outstanding properties [2] as it has an excellent combination of mechanical and physical properties [3]. Amid all Al alloys, Al 6061 possesses outstanding formability besides its high strength and excellent corrosion resistance [4, 5]. The superior performance of nanocomposites can be obtained by uniform, homogeneous and embedding of nanoparticles over the nanocrystallite matrix [6]. Agglomeration/clustering of very fine/nanoreinforcement particles over metal matrix would deteriorates the mechanical properties [7]. However, it can be avoided by mechanical alloying (MA) which is one of the solid-state powder metallurgy (P/M) process [2]. Further, uniform distribution and embedding of nanoreinforcement over the matrix can be achieved which would improve the mechanical performances of materials. The mechanical alloying is one of the severe plastic deformation (SPD) technique. During mechanical alloying (MA), high strain is given on the material and the structural refinement such as crystallite size reduction, lattice strain increment, changes in dislocation density, lattice parameter variation etc., would occur by shear and fracture of powder phase mixtures. By this process, nanostructured/nanocrystallite materials can be manufactured [2]. Conventional cold uniaxial die compaction process is one of simple and economic one of consolidation of powder materials into bulk-shaped products followed by sintering under controlled atmosphere. Various authors have worked on P/M aluminum-based metal matrix composites reinforced by ceramic particles such as graphite, silicon carbide particulate (SiCp), aluminum oxide (Al₂O₃), titanium carbide (TiC), vanadium carbide (VC), aluminum nitride (AlN), boron carbide (B₄C), silicon nitride (Si₃N₄), titanium boride (TiB₂), aluminum boride (AlB₃), zirconium carbide (ZrC) and magnesium boride (MgB₂) which have been successfully dispersed and investigated via mechanical alloying/mechanical milling. There is no detailed study concerning TiO₂ powders used as a reinforcement in Al-based MMCs. Titania (TiO₂) has outstanding properties, namely, good wear and corrosion resistance, low coefficient of thermal expansion, high thermal shock resistance, excellent catalytic performance and thermal stability at high temperatures [8, 9]. The material selected as AA 6061-TiO₂ particulate MMCs is mainly proposed for automotive and aircraft parts. For instance, it can be suitable for automotive engine pistons and connecting rod, bicycle hub, bike frames, valves and valve parts, brake pistons, hydraulic pistons, marine fittings, electrical fittings, contactors, aircraft fittings and couplings.
2. Synthesis and consolidation procedure of nanocomposites

2.1. Nanocomposite powders preparation

In the present investigation, the AA 6061 Al alloy reinforced with different weight percentage of TiO$_2$ particulate nanocomposite powders was prepared by MA (high-energy ball milling) method. The AA 6061 Al alloy matrix composition and powder size of each elemental powder used in this study are given in Table 1. Figure 1(a) and (c) shows the morphologies of as-received Al and TiO$_2$ powder particles, respectively, using secondary electron image (SEI) of scanning electron micrograph (SE-SEM). From Figure 1(a) and (c), it can be observed that the Al matrix powder particles were in irregular flake-like shape and TiO$_2$ particles were in clustered tetragonal shape. Figure 1(b) and (d) shows the X-ray diffraction patterns of as-received Al and TiO$_2$ particles, respectively, which indicated the well-crystalline nature of the powders. The as-received TiO$_2$ particles are in anatase formed and tetragonal structure with average clustered particles size of <1 μm which was measured by laser scattering system technique. The prepared nanocomposites contained 0, 2, 4, 6, 8, 10 and 12 wt.% of TiO$_2$ powder particles were completely embedded in the soft alloy matrix using the planetary type high-energy ball mill (Insmart systems Ltd., Hyderabad, India) (Figure 2a). Nine highly hardened stainless steel balls of 20 mm diameter (33.5 g mass of each ball, totally, 301.5 g) together with 30 g mass of AA 6061$_{100-x}$–x wt.% TiO$_2$ (x=0–12 with a step of 2 wt.%) powder mixture under toluene medium as a process control agent (PCA) were poured in a hardened stainless steel vial. The ball-to-powder ratio (BPR) was set approximately 10:1. The plate (i.e., sun wheel) speed of mill was set to 100 rpm (bowl/vial speed, 280 rpm). The milling was carried out in wet medium using toluene ($C_6H_5CH_3$) of sulfur free (Ranbaxy, India) to prevent undue oxidation, agglomeration of powders, balls and vial with the powder [10]. The composite powders containing the different wt.% of TiO$_2$ were milled up to 40 h. The milling was carried out up to 20 min and then alternated with 20 min of cooling to avoid significant temperature rise. The

<table>
<thead>
<tr>
<th>Name of the element(s)</th>
<th>Purity, %</th>
<th>Elements concentration (gravimetric, wt.%)</th>
<th>Powder size, µm (mesh size)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicon, Si</td>
<td>99.3</td>
<td>0.600</td>
<td>-45 (&lt;325)</td>
</tr>
<tr>
<td>Iron, Fe</td>
<td>99.7</td>
<td>0.700</td>
<td>-75 to +45 (&lt;200 to &gt;325)</td>
</tr>
<tr>
<td>Copper, Cu</td>
<td>99.4</td>
<td>0.275</td>
<td>-45 (&lt;325)</td>
</tr>
<tr>
<td>Manganese, Mn</td>
<td>99</td>
<td>0.150</td>
<td>-75 to +45 (&lt;200 to &gt;325)</td>
</tr>
<tr>
<td>Magnesium, Mg</td>
<td>99.7</td>
<td>1.000</td>
<td>-75 to +45 (&lt;200 to &gt;325)</td>
</tr>
<tr>
<td>Chromium, Cr</td>
<td>99.8</td>
<td>0.195</td>
<td>-45 (&lt;325)</td>
</tr>
<tr>
<td>Zinc, Zn</td>
<td>99.4</td>
<td>0.250</td>
<td>-45 (&lt;325)</td>
</tr>
<tr>
<td>Titanium, Ti</td>
<td>99.3</td>
<td>0.150</td>
<td>-45 (&lt;325)</td>
</tr>
<tr>
<td>Aluminum, Al</td>
<td>99.7</td>
<td>Bal</td>
<td>-45 (&lt;325)</td>
</tr>
</tbody>
</table>

Table 1. Chemical composition and powder size used to make AA 6061 Al alloy matrix.
Figure 1. The morphology of as-received powders: (a) Al and (c) TiO₂. XRD patterns of as-received powders: (b) Al and (d) TiO₂.

Figure 2. (a) High-energy planetary ball mill and (b) schematic diagram showing the synthesis of nanocomposite powders.
schematic diagram of synthesizing the nanocomposite by high-energy ball milling method is also shown in Figure 2b.

2.2. Synthesis of trimodeled composite powders

In order to evaluate the improvement of ductility while maintaining high strength and toughness for nanostructured AA 6061-TiO$_2$ composite materials, trimodeled AA 6061-TiO$_2$ nanocomposite powders (three phases consisting of nanocrystallite matrix, nano ceramic and coarse crystallite matrix) were synthesized by the same planetary high-energy ball milling. For instance, first, the nanostructured composite powder of AA 6061-12 wt.% TiO$_2$ was synthesized by 40 h of MA using the same ball milling parameters mentioned in Section 2.1 (consists of nanocrystallite matrix and nano-TiO$_2$ ceramic). Then, these nanocomposite powders were mechanically blended with 0, 5, 10, 15, 20, 25 and 30 wt.% coarse grains (CG) elemental powders corresponding to AA 6061 alloy matrix composition in the same planetary ball mill. Here, the BPR of 1:1 at 120 rpm for 2 h was carried out.

2.3. Consolidation of composite powders

A hydraulic press (Figure 3a) capacity of 40 tons made by Insmart systems, Hyderabad, India, was used to do uniaxial compaction. The cold uniaxial compaction is an important process in P/M to attain near net shape fabrication of engineering components prior to sintering [11, 12]. First, the precursor powders obtained from high-energy ball milling were cold compacted using conventional cold uniaxial pressing in a rigid double end compaction type with compaction pressure of 500 MPa [13, 14]. The obtained dimensions of compacted samples called green pellets were 10 mm in diameter and 6–7 mm thickness [15]. The die was made of high-carbon high-chromium hardened and tempered steel (inner and outer diameters of 10 and 60 mm), and the two punches (10 mm diameter) were made of the same material as die to consolidate the powders from top to bottom simultaneously. Zinc stearate [Zn(C$_{18}$H$_{35}$O$_2$)$_2$] was used as lubricant to minimize the friction between powder particles and die wall during compaction process. Figure 3b shows the schematic design of the double end compaction die.

For investigating the sintering behavior, the consolidated nanocomposites of AA 6061$_{100-x}$–x wt.% TiO$_2$ (x=0, 4, 8 and 12 wt.%) of the green pellets compacted at 500 MPa were degassed first at 350°C for 60 min [16]. Then, the degassed samples were sintered for 120 min in the temperature range of 400, 475, 550 and 625°C under reducing atmosphere [17] in a mechanical pusher furnace as applicable to P/M industries. The schematic diagram of mechanical pusher furnace is shown in Figure 4.

2.4. Consolidation of trimodeled composite powders

The synthesized trimodeled AA 6061-12 TiO$_2$ nanocomposite consisting of 0, 5, 10, 15, 20, 25 and 30 wt.% CG 6061 alloy (obtained from elemental powders by mixing) matrix powders was first dried and stress recovered at 343 K under N$_2$ atmosphere (mass flow rate of 63 l/h and dew point temperature of <40°C) [17]. The stress recovering of trimodeled composite powders was carried out to avoid the unwanted oxide phase at elevated temperature during sintering. These stress recovered powders were then consolidated by cold uniaxial compaction at 500 MPa. The post-compacts were degassed and then sintered at 823 K for 6 h under N$_2$ atmosphere [17].
3. Structural characterization methods

3.1. Geometric characterization techniques

The transmission electron microscope (TEM) and X-ray diffraction (XRD) can be effectively used for investigating the structural characterization of fine powders, polycrystalline materials, amorphous materials and thin films. These two techniques are the commanding
3.2. X-ray diffraction (XRD)

The XRD patterns are used to identify the structure and the different phases present in the materials. The phase constitution of milled powders, post-compacts, post-sintered and post-deformed preforms are evaluated by XRD using CuKα radiation. The used XRD equipment is made by D/MAX ULTIMA III, Rigaku Corporation, Japan, operating at 30 mA and 40 kV. The set 2°/min scanning speed and 20–100° scanning range were used here. It is well known that the average crystallite size and lattice strain or microstrain or lattice distortions owing to the presence of lattice defects which commonly occurred in MA products can be easily measured by XRD peaks of line profile analysis [20]. During MA with respect to milling time and percentage of reinforcements in the matrix, XRD peak broadening occurs which depends upon crystallite size and strain present in the nanocomposites/nanomaterials. Therefore, nanostructured materials/nanocomposites have a large volume of grain boundaries when compared to bulk materials [21]. If a material is not having any strain mean, Scherer’s formula can be used to calculate crystallite size (Simplest method) [22]. Williamson–Hall (W–H) [23] and Warren–Averbach (W–A) [24] analyses are other two analytical methods which are generally used to measure the strain and the crystallite size of nanomaterials/nanocomposites. The W-A method is based on Fourier analysis of peak intensities in which Fourier coefficients are used to separate the crystallite size and strain contribution from XRD peak profiles. However, this approach needs peak intensities at high diffraction angles. But, it is difficult to use this method when higher angle reflections are weak [25]. On the other hand, the W–H method is used to investigate the same information at lower diffraction angle. Also, it is easy and suitable for lattice strain and crystallite size calculation of cubic crystals. Therefore, reduction in crystallite size and rise in microstrain can be found out using W-H analysis of Eq. (1).

\[
\beta_{hkl} \cos \theta_{hkl} = \left( \frac{K \lambda}{T} \right) + 4 \varepsilon \sin \theta_{hkl}.
\]

Here, K is the shape factor of 0.9, λ is the X-ray wavelength of 1.5406 Å, θ_{hkl} is the Bragg angle in degree and t is the effective crystallite size in nm and ε is the lattice strain. The X-ray line profile breadth, \( \beta_{hkl} \), after corrected instrumental broadening as a full width at half-maximum (FWHM), was calculated on each reflection of 2θ. For constructing a linear plot of \( \beta_{hkl} \cos \theta_{hkl} \) against 4sin\( \theta_{hkl} \) for determining crystallite size and lattice strain, X-ray diffraction of first five reflections (1 1 1), (2 0 0), (3 1 1), (2 2 2) and (4 0 0) was used. Then, crystallite size (t) was obtained from the intercept c (i.e., c=Kλ/t) and the strain (ε) from the slope (i.e., m=ε).

3.3. Scanning electron microscope (SEM)

The milled powder samples were analyzed for powder morphological evaluation by scanning electron microscopy (SEM) using HITACHI S 3000 H operating at 8.5 mA and 20 kV.
Before examining the powder samples in SEM, sputtering was done on the surface of the powders to conduct charge away from the surface. The microstructure of sintered preforms (after polishing and etching) was examined by F E I Quanta FEG 200—high-resolution scanning electron microscope (HR-SEM) with EDAX operating at 30–50 kV. In order to identify and confirm the elements shown in the SEM micrographs, energy dispersion spectrometer (EDS) was used. During analysis, both secondary electron image (SEI) mode and back-scattered electron image (BSEI) mode were operated.

3.4. Transmission electron microscope (TEM)

To measure the matrix crystallite size exactly, and identifying the embedding and distribution of nano level reinforcement in the matrix grain, both dark and bright field image of transmission electron microscope (TEM) can be used. Selected area of diffraction (SAD) patterns is also used to confirm the NC nature of the samples. Here, PHILIPS CM 12 TEM was used with spot EDAX facility. For powder samples, first the ball-milled powder particles were under suspension in ethanol (volatile liquid) and then a drop of this suspension was allowed to dry on a thin carbon foil supported by a conventional microscope grid leaving the powder particles ready for the observation. For sintered solid samples, manual polishing and mechanical grinding were performed until the sample thickness <100 Åμm was achieved. Following mechanical grinding, the samples were then dimpled to sample thickness of approximately 50 Åμm. Finally, ion milling was carried out on the dimpled samples using an ion miller of Edwards Model E306A.

4. Mechanical testing methods

4.1. Density measurement

The density of the post-compacts and sintered preforms was estimated precisely by Archimedes principle [26] using electronic density balance. This principle is based on first measuring weight of sample in air column and then weighed in water column. Then, the ratio of weight of sample in air column and apparent loss of weight (difference between weight of air column and water column) give the specific gravity (SG) of post-compacts/sintered preforms. Lastly, the density of post-compact/sintered density can be determined by multiplying the SG and density of water. Here, paraffin treatment was performed over the samples to avoid infiltration of water into the sample [27]. The calculated error in the density treatment was conformed within 1%. Three independent experimental readings were used and averaged for investigation. In addition, theoretical density of sintered composites with different percentage of reinforcements was determined using the rule of mixture.

4.2. Hardness measurement

Mechanical properties in terms of strength can be analyzed using hardness measurements. The hardness of sintered preforms was measured using PC-based Ratnakar Vickers tester. Here, the hardness was tested at a load of 1 kg. At least 15 measurements were taken.
in various places of polished samples for each sample. Then the average was taken for investigation.

4.3. Simple compression test

The uniaxial simple compression test was done using servo-controlled universal testing machine with strain rate of $10^{-3}$ s$^{-1}$ at room temperature to determine the maximum compressive strength. For compression test, the sample size of 5 mm×5 mm×10 mm was used as per ASTM standard. The samples were cut from sintered one by wire-cut electrical discharge machine; then polished by abrasive sheet of SiC paper to 1200 grit; further graphite lubricant was applied at the top and bottom surface of samples to minimize friction effect. Here, three samples were used in each composition, and the average was used for the interpretation.

5. Results and discussion

5.1. Morphology of nanocomposite powders as function reinforcement

The main purpose of studying the powder surface morphological examination is to investigate the powder particle shape changes while manufacturing of AA 6061$_{100-x}$-x wt.% TiO$_2$, x=0, 2, 4, 6, 8, 10 and 12 wt.% particulate nanocomposite powders produced by MA. The homogeneous distribution of reinforcement particles over the nanocrystallite matrix, embedding of reinforcement particles inside the nanocrystallite matrix, presence of any agglomeration or clustering of reinforcement particles with the nanocrystallite matrix, powder particles shape and particle size can be investigated through the ball-milled powder particles as it influences the mechanical properties. The powder morphology can be evaluated qualitatively by secondary electron image (SEI) of SEM and back-scattered electron image (BSEI) of SEM. Figure 5 shows the influence of reinforcement addition in the nanocrystallite matrix powders after 40-h MA. It can be clearly seen from Figure 5 that the size of the particle/agglomerate decreased with the function of reinforcement considerably. However, almost spherical-shaped and equiaxed particles were obtained in all the system with the same milling condition (40 h). These results implied the attainment of equilibrium state after 40-h MA. The morphological results showed that the addition of TiO$_2$ particles in the matrix produced the powder morphology size reduction of matrix steadily. This was attributed to TiO$_2$ ceramic particles also acted as milling agent. Further, it was observed here that the embedding and dispersing of nano-sized TiO$_2$ particles of around 119 nm with the soft Al alloy matrix powder for AA 6061-12 wt.% TiO$_2$ nanocomposite (Figure 5h). The morphological results showed that the addition of TiO$_2$ particles favors to refining of matrix particles during MA. On the other hand, despite the ductile nature of the Al matrix, the formation of large agglomerate size (Figure 5a) was diminished with the presence of TiO$_2$ particles. This is due to the fact that the increasing percentage of TiO$_2$ particles in the soft matrix decreases the ductility so as to increase the amount fracture in the matrix particles before impacts of matrix particles that cause lamination. These results inferred here that more amount of work hardening occurred in higher amount of reinforcement. The decreasing of particle/agglomerate size of the matrix with carrying reinforcement was also observed by several
Figure 5. The morphology of AA 6061, x wt.% TiO$_2$ nanocrystallite/nanocomposite powder after 40-h MA: (a) 0%, (b) 2%, (c) 4%, (d) 6%, (e) 8%, (f) 10%, (g) 12% and (h) BSEI of magnified view of (g) shows the embedding of TiO$_2$ particles on the matrix.
researchers related to Al-based MMCs processed via MA/MM [33–36]. Figure 6 shows the X-ray mapping of 40-h powders which confirms the uniform TiO$_2$ particles within the Al matrix (Red: Al, Green: O and Blue: Ti).

5.2. Effect of percentage of reinforcement on powder morphology of nanocomposite powders

Figure 7 shows the influences of the addition of TiO$_2$ ceramic particles on nanocrystallite AA 6061 matrix particles size. It was observed here that the intensity of fracture mechanism increased and dominated the soft ductile matrix particle-particle cold welding as the percentage of TiO$_2$ ceramic particles increased in the soft Al alloy matrix. These resulted the formation of very fine particles/agglomerates in the case of 12 wt.% TiO$_2$-reinforced nanocomposites (Figures 5g and 7) when compared to unreinforced nanocrystallite alloy powder (Figures 5a and 7) during the same milling condition (40 h). The increasing of hard ceramic particles would accelerate the fracturing process which was also reported elsewhere [37]. It was attributed to importing more collisions on matrix powders which meant high-velocity energy acted over the matrix powders. Thus, it was clear here that the tendency for fracturing was higher than the cold welding for longer milling time of 40 h. In general, after reaching the steady state of longer milling (40 h here) under wet milling, particle-particle cold welding on soft ductile alloy matrix would occur. Consequently, the particle/agglomerate size would start to increase (Figure 5a) when compared to un-milled matrix powder (Figure 1a). This was attributed to higher cold welding tendency overcoming the fracture process [38]. However, almost equiaxed with spherical-shaped and refined microstructure were obtained. Further, the rate of soft alloy matrix particle-particle getting cold-welded started to decrease as the percentage of TiO$_2$ ceramic particles increased. This was attributed to more domination of fracturing tendency of TiO$_2$ particles. Therefore, the powder/agglomerate size started to decrease in the case of higher reinforced nanocomposites (AA 6061-12 wt.% TiO$_2$, Figures 5g and 7). Similar kind of results was observed by Fogagnolo et al. 2003 [39] during ball milling.

![Figure 6](image_url)
of Al-AlN system. Also, it can be explained in another way that the strength increased as the percentage of reinforcement particles increased according to Orowan strengthening mechanism.

The particle size of reinforcement phase decreased in which the clustered average particle size of TiO$_2$ of around 1 μm (Figure 1c) was reduced to individual TiO$_2$ particles of 119 nm (Figure 5h). The TiO$_2$ particle size after 40-h MA of AA 6061-12 wt.% TiO$_2$ nanocomposite powders was checked using bright and dark field image of transmission electron microscope (TEM) (Figure 8). Using several bright and dark field images of TEM, the average TiO$_2$ particle size of around 120 nm was obtained (50 particles were counted), which coherent with the results obtained from back-scattered electron image of SEM morphology. The pink color arrow head indicates the TiO$_2$ particles. Hence, the result of the higher percentage of TiO$_2$ phase in the soft matrix produced smaller particle/agglomerate size of the nanocomposite powders (40-h milling) (Figure 5g) [39–41]. The addition of TiO$_2$ powder particles had great influence on the morphological characteristics of the fabricated nanocomposite powders via MA.
5.3. Structural evaluation of mechanically alloyed nanocomposite powders

The process of manufacturing of MMCs powders through MA causes morphological and structural changes. Severe plastic deformation of the powder particles during MA can lead to grain refining, variation in the crystallite size, accumulation of internal stress, density of dislocation and variation of the lattice parameter [42]. The objective of this study is to report the structural characteristics of MAed powders (40 h) as the function of reinforcement (0, 2, 4, 6, 8, 10 and 12 wt.% TiO$_2$) using XRD, TEM and EDS.

5.3.1. XRD analysis

Figure 9 shows the XRD patterns of Al 6061–x wt.% TiO$_2$ (x=0, 2, 4, 6, 8, 10 and 12 wt.%) nanocomposites powders after 40-h MA. This figure clearly shows that the Al peak width, for example, at (1 1 1) plane, increased with the percentage of reinforcement. This was due to which TiO$_2$ particles were also acting as a milling agent which accelerated the milling process. The inset of Figure 9 shows that the case of 12 wt.% TiO$_2$ nanocomposite powders more broadened Al peak at (1 1 1) plane occurred than unreinforced nanocrystallite powders. It indicates that more spatial coherent length confinement occurred in higher reinforced nanocomposite [43]. Also, it can be noted from the inset of Figure 9 that shifting of Bragg’s angle occurred in the case of 12 wt.% TiO$_2$ particulate nanocomposite powders. These results show that more distortion occurred in higher reinforced nanocomposite powder than unreinforced nanocrystallite powder during the same milling time. Further, the shifting of Bragg’s angle indicates the changes in the lattice parameter value with respect to percentage of reinforcement during 40-h MA. The increasing of peak broadening of matrix phase by the addition of reinforcement ceramic phase can be clearly seen from Figure 10. The structural characteristics such as crystallite size, lattice microstrain, dislocation density, lattice parameter and the unit cell volume of Al as the function of reinforcement after 40-h milling are reported in Table 2. Owing to the characteristics of high-energy ball milling, large amount of structural defects in terms of dislocations would occur due to severe plastic deformation [44]. Severe plastic deformation (SPD) of high-energy milling brings about a deformed lattice with high dislocation density during early stage of milling. These indicate the amount of dislocations pile up near the GBs.
will be increased. When prolonged milling, sometimes the GBs might have turned to be soft or relaxed. On this time, the amount of dislocations pileup near the GBs will be decreased. Hence, for MAed powders, dislocations are the main defects of which dislocation density \( \rho_D \) can be determined as [45]:

\[
\rho_D = 2 \sqrt{3} \frac{\langle \varepsilon_{\text{r.m.s.}} \rangle}{tb},
\]

(2)

where \( \langle \varepsilon_{\text{r.m.s.}} \rangle \) is RMS lattice strain which is determined as:

\[
\langle \varepsilon_{\text{r.m.s.}} \rangle = \left( \frac{2}{\pi} \right)^{1/2} \frac{\Delta d}{d_{(hkl)}}
\]

(3)

\( \Delta d = d_{(hkl)} - d_{0(hkl)} \) where \( d_{(hkl)} \) and \( d_{0(hkl)} \) are the calculated and observed interplanar spacing, \( t \) is the crystallite size in m and \( b \) is the burgers vector of dislocation in m which is equal to \( a/\sqrt{2} \) for the FCC structure, \( a \) is the lattice parameter in m. The actual lattice parameter was

Figure 9. XRD patterns of AA 6061_{100-x}–x wt.% TiO\(_2\) (x=0, 2, 4, 6, 8, 10 and 12%) nanocrystallite/nanocomposite powder after 40 h of high-energy ball milling. Inset shows shift in Bragg's angle.
obtained as described by Cullity [22], by constructing the linear plot between the calculated lattice parameter for each Bragg’s angle and the corresponding value of \( \cos^2 \theta / \sin \theta \).

### 5.3.2. TEM analysis

The TEM micrographs of AA 6061\(_{100-x}\)-x wt.% TiO\(_2\) (x=0, 4, 8 and 12%) nanocomposite powder is shown in Figure 11a–l. The matrix crystallite size was calculated based on several bright and dark field images (Figure 11), and minimum of more than 200 grains was counted.

<table>
<thead>
<tr>
<th>Composition</th>
<th>Grain size ((d)), nm</th>
<th>Lattice strain ((e)), %</th>
<th>r.m.s. strain ((q) (\times 10^4)), m(^2)</th>
<th>Dislocation density ((q) (\times 10^3)), m(^2)</th>
<th>Lattice parameter ((a)), Å</th>
<th>Unit cell volume (V), Å(^3)</th>
<th>Volume fraction of TiO(_2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6061 AA</td>
<td>65±2.50</td>
<td>0.325±0.0031</td>
<td>2.5961</td>
<td>4.847±0.0025</td>
<td>4.047±0.00020</td>
<td>66.297±0.0098</td>
<td>0</td>
</tr>
<tr>
<td>6061 AA+2% TiO(_2)</td>
<td>60.5±1.85</td>
<td>0.164±0.0018</td>
<td>1.3082</td>
<td>2.615±0.0015</td>
<td>4.047±0.00015</td>
<td>66.287±0.0042</td>
<td>0.01793</td>
</tr>
<tr>
<td>6061 AA+4% TiO(_2)</td>
<td>58±5.00</td>
<td>0.190±0.0024</td>
<td>1.5214</td>
<td>3.198±0.0078</td>
<td>4.047±0.00015</td>
<td>66.287±0.0074</td>
<td>0.03588</td>
</tr>
<tr>
<td>6061 AA+6% TiO(_2)</td>
<td>53±3.2</td>
<td>0.149±0.0020</td>
<td>1.1893</td>
<td>2.562±0.0032</td>
<td>4.047±0.00018</td>
<td>66.297±0.0035</td>
<td>0.04937</td>
</tr>
<tr>
<td>6061 AA+8% TiO(_2)</td>
<td>50±4.60</td>
<td>0.223±0.0017</td>
<td>1.7805</td>
<td>4.365±0.0034</td>
<td>4.046±0.00020</td>
<td>66.286±0.0045</td>
<td>0.06587</td>
</tr>
<tr>
<td>6061 AA+10% TiO(_2)</td>
<td>48.7±2.5</td>
<td>0.216±0.0016</td>
<td>1.7276</td>
<td>4.263±0.0021</td>
<td>4.046±0.00024</td>
<td>66.286±0.0022</td>
<td>0.07896</td>
</tr>
<tr>
<td>6061 AA+12% TiO(_2)</td>
<td>46±2.00</td>
<td>0.199±0.0023</td>
<td>1.5903</td>
<td>3.940±0.0019</td>
<td>4.046±0.00015</td>
<td>66.272±0.0031</td>
<td>0.09862</td>
</tr>
</tbody>
</table>

Table 2. Structural characterization of AA 6061\(_{100-x}\)-x wt.% TiO\(_2\) composite powder after 40-h milling, x = 0, 2, 4, 6, 8, 10 and 12%.

Figure 10. XRD patterns of AA 6061\(_{100-x}\)-x wt.% TiO\(_2\) x=0, 4, 8, and 12%, composite powder after 40 h of high-energy ball milling.
Figure 11. TEM micrographs of as-milled nanocomposite powders: (a) bright field image (BFI) of 0% TiO$_2$, (b) SAD pattern of 0% TiO$_2$, (c) EDAX analysis of 0% TiO$_2$, (d) BFI of 4% TiO$_2$, (e) dark field image (DFI) of 4% TiO$_2$ (inset shows the SAD), (f) EDAX analysis of 4% TiO$_2$, (g) BFI of 8% TiO$_2$, (h) DFI of 8% TiO$_2$ (inset shows the SAD), (i) EDAX analysis of 8% TiO$_2$, (j) BFI of 12% TiO$_2$, (k) DFI of 12% TiO$_2$ (inset shows the SAD) and (l) EDAX analysis of 12% TiO$_2$. Note: single arrow represents TiO$_2$ particle.

for each composition to get the average grain size. The calculated matrix crystallite size for 0, 4, 8 and 12 wt.% TiO$_2$ nanocomposite powders was to be around 69, 63, 54 and 49 nm, respectively. From the selected area of diffraction (SAD), the continuous sharp ring pattern was observed in all the system which confirmed the nanocrystalline nature of the Al matrix in as-milled (MA, 40 h) condition [34]. Single arrow in the bright field images of Figure 11 indicates the TiO$_2$ particle embedded in the matrix. Further, the corresponding EDAX analysis was also performed and tabulated in the respective figures. The presence and embedding
of TiO\textsubscript{2} particles in the matrix were confirmed by EDAX spectrum in which the TiO\textsubscript{2} spectrum was observed to increase with percentage of reinforcement.

5.3.3. Effect of percentage of reinforcement on structural changes

The XRD results from Figure 10 corroborate that an increase in the amount of TiO\textsubscript{2} in the soft matrix results in a finer crystallite size. The crystallite size variation with percentage of reinforcement is shown in Figure 12. The crystallite size of soft matrix was around 65, 60, 57, 53, 50, 48 and 46 nm for 0, 2, 4, 6, 8, 10 and 12 wt.% of TiO\textsubscript{2} particulate composite powder, respectively, after 40-h MA. As the weight percentage of hard TiO\textsubscript{2} content increased, the ductility of the alloy matrix particles decreased, resulting in the dominance of fragmentation which led to a finer grain size in the case of higher TiO\textsubscript{2} content. The estimated lattice parameter (Table 2 and Figure 12) of Al matrix for all reinforced nanocomposite powder was a lower value than the lattice parameter of pure FCC-Al at room temperature (4.0496 Å). This indicates that distortions occurred in the Al lattice and decreasing of lattice parameter with percentage of reinforcement meaning that ceramic phase addition influenced the lattice parameter considerably [46]. The decreased lattice parameter with percentage of reinforcement was due to the balancing between the dissolution of Ti and O atoms in which O atoms dissolution dominated at the higher reinforcement particulate composite powders. Because, the atomic radius of O atom is lower than the atomic radius of Al atom and the atomic radius of Ti atom is higher than Al. Therefore, the O atom can easily dissolve in the Al lattice which decreased the lattice parameter. However, numerically, the deviation of lattice parameter value with percentage of reinforcement was very little. This can be checked by calculating

![Figure 12](http://dx.doi.org/10.5772/65947)
the percentage of solid solution of TiO$_2$ particles from XRD results. The percentage of solid solution of TiO$_2$ increased with the percentage of reinforcement. Numerically, this value is equivalent to zero.

5.4. Sintering behavior of nanocomposites

The excellent mechanical properties can be expected from P/M processed composites when the second phase particles (here TiO$_2$ ceramic particles) is homogeneously distributed in the matrix. Generally, in conventional metal matrix composites (MMCs), matrix-to-reinforcement particle size ratio would affect the mechanical performances of MMCs. If matrix-to-particle size ratio is greater than unity mean, clustering/agglomeration of reinforcement particles over the matrix would occur which decrease the mechanical properties. This is owing to the fact that reinforcement clusters/agglomerates cannot transfer tensile and shear stresses as it form interparticulate boundaries that act cracks [46]. Therefore, the main objectives of the present section are to investigate whether there is any effect on matrix-to-reinforcement particle size ratio (here, all the samples are greater than unity) as here the reinforcement particles size is around 119 nm, percentage of reinforcement and grain refinement on hardness and densification behavior of nanocomposites.

5.4.1. Influence of reinforcement content on sintering of nanocomposites

Figure 13a shows the densification in terms of percentage of theoretical density of nanocomposite sintered over the temperature range 400–625°C with a step size of 75°C. The percentage of theoretical density sintered nanocomposite at 400°C was 80.25, 81.50, 83.00 and 84.50% for 0, 4, 8 and 12 wt.% TiO$_2$ particles, respectively, which varied from 80 to 84%. It is interesting to note that the percentage of theoretical density was increased from 80% (unreinforced NC/UFG alloy) to 84% (12 wt.% reinforced nanocomposite) with the function of reinforcement. The increasing percentage of theoretical density with the function of reinforcement was attributed to abrupt changes in the powders morphology size reduction. The observed rate of increased percentage theoretical density was marginally greater up to 4% TiO$_2$, and then, it increased considerably up to 12 wt.% TiO$_2$. This was attributed to a powder matrix particle size reduction, uniform distribution and complete embedded/dispersed TiO$_2$ particles on the soft alloy matrix. The sintering response with function of reinforcement and function of temperature can be evaluated by a contour graph which is shown in Figure 13b. From Figure 13b, the percentage of theoretical density curves increased steadily first over both the percentage of reinforcement and sintering temperature up to 500°C. Beyond the sintering temperature of 550°C, irrespective of the reinforcement, the percentage of theoretical density increased slightly. One can notice that the percentage of theoretical density increased with the function of reinforcement. The highest percentage of theoretical density can be achieved when the percentage of reinforcement is >10 wt.% between the temperature range from 500 to 550°C. The highest percentage of theoretical density achieved on this region was attributed to powder morphology size reduction and homogeneous with embedded nano-sized Titania particles in the matrix [47]. Further, at the lower value of percentage of reinforcement (<2 wt.%), the percentage of theoretical density can be achieved only around 80%. This worsened sintering
behavior was attributed to very large agglomerate size with almost spherical morphology of the matrix after 40-h milling (Figure 5a).

5.4.2. Structural evolution of sintered nanocomposites

The XRD patterns of 0, 4, 8 and 12 wt.% reinforced particulate nanocomposite sintered at 550°C for 2 h are shown in Figure 14. The observed phases are mainly α-Al and TiO$_2$. The crystallite size of α-Al phase for these sintered nanocomposites was calculated by standard Williamson Hall analysis. The calculated crystallite size after sintering at 550°C is 308, 265, 238 and 205 nm for 0, 4, 8 and 12 wt.% reinforced nanocomposite, respectively. The measured crystallite sizes were in ultrafine level. To confirm the crystallite size in ultrafine level, AA 6061–12 wt.% TiO$_2$ sintered at 550°C sample was examined using TEM analysis. Figure 15a shows the bright field image of 12 wt.% reinforced nanocomposite. From Figure 15a, the observed crystallite size of α-Al matrix was almost equiaxed. Further, nanometer-sized Titania particles were uniformly distributed and embedded in the α-Al matrix. Figure 15b shows the corresponding selected area of diffraction (SAD) patterns which produced a ring pattern. These results confirmed that the individual grains of α-Al matrix were separated by high-angle grain boundaries and have a random orientation with neighboring grains. Also, the SAD ring pattern reveals that α-Al grains are in ultra-fine level. Further, the average grain size of the α-Al matrix is found to be around 225±15 nm based on 250 grains counted from the bright field images of TEM microstructure.

Figure 16a shows the crystallite size of AA 6061$_{100-x}$–x wt.% TiO$_2$ (x=0, 4, 8 and 12%) nanocomposites in as-milled and as-sintered at 550°C. Metal matrix composites are all immiscible systems in which the diffusion (migration of atoms) of one kind into the other is usually restricted because crystallite growth would occur based on diffusion. Therefore, MMCs, in such immiscible systems very small crystal growth is expected on heating [48]. This is...
clearly evident from Figure 16a, wherein the growth of crystallite size of α-Al was restricted significantly after sintering at 550°C for higher percentage of reinforcement. It is to be noted here that an increase in the TiO$_2$ content of the nanocomposites, the nano-sized Titania (∼119 nm) particles pinned the matrix grain boundary and prevented the grain growth dur-
ing sintering. An increase in the TiO$_2$ content reduced the probability of Al-Al contacts and increased the probability of Al-TiO$_2$ interfaces and hence reduced their tendency for crystallite growth due to the immiscible nature of the soft Al alloy matrix with hard TiO$_2$ particles [49].

5.4.3. Influence of reinforcement content on hardness of sintered nanocomposites

The measured Vickers hardness values as function of sintering temperature is given in Table 3. From Table 3, it was observed that the Vickers hardness values increased steadily first up to 4% TiO$_2$ and then increased abruptly as the percentage of reinforcement increased after 40-h milling. The highest value of hardness for AA 6061-12 wt.% TiO$_2$ nanocomposites was obtained after sintering at 625°C. This was attributed to crystallite refinement occurred in the structure, very fine particle size of matrix and embedding of larger quantity of TiO$_2$ particles in the matrix [46]. Figure 16b shows the variation in hardness of the nanocomposites sintered at 625°C as a function of percentage of TiO$_2$ particles. The hardness varied from 564 to 1126 MPa to the corresponding increase of TiO$_2$ content. This higher value was attributed to their finer grain size and the presence of nano-sized TiO$_2$ particles as dispersoid in the matrix and embedded in the soft matrix. Very high hardness value was obtained at higher reinforcement [46], which was due to very fine particle size distribution, crystallite refinement, highly embedded TiO2 ceramic particles in the matrix and improved dislocation density at the grain boundary. The hardness of AA 6061-12 wt.% TiO$_2$ nanocomposites was 1.41–2.11 times higher than that of unreinforced AA 6061 unreinforced alloy. For instance, the observed size of the hardness indentation for 0, 4, 8 and 12 wt.% TiO$_2$ nanocomposite sintered at 550°C was around 0.185, 0.179, 0.161 and 0.146 μm, respectively. The matrix crystallite sizes based on peak profile analyses were 308, 265, 238 and 205 nm (Figure 14) for 0, 4, 8 and 12 wt.% TiO$_2$ nanocomposite, respec-

Figure 16. (a) Crystallite size as function of reinforcement in as-milled and as-sintered at 550°C condition and (b) effect of composition on Vickers hardness of AA 6061$_{100-x}$-x wt.% TiO$_2$ (x=0, 2, 4, 6, 8, 10 and 12) bulk micro and nanocomposites (sintered at 625°C).
tively. These results indicated that as the crystallite size decreases, the hardness impression was also decreases steadily due to more refinement occurs in the structure with the reinforcement content in the same milling condition. It can be concluded here that there was no possibility of clusters/agglomerates formation in these nanocomposites; the matrix-to-reinforcement particle size ratio was insignificant in Vickers hardness. To conform this one, the back-scattered electron images (BSEI) of 2, 6 and 8 wt.% TiO$_2$-reinforced particulate sintered nanocomposite at 550°C is shown in Figure 17a–f. In this, left side of Figure 17a, c and e shows the microstructure of sintered preform at low-magnification (400 ×) value. The corresponding magnified view is shown on right side of Figure 17b, d and f for 2, 6 and 8 wt.% reinforced composite, respectively. Even at high magnification itself in all preforms, there was no clustering observed in the present manufactured nanocomposite. In Figure 17, double arrow represents the distribution of reinforcement particles on the matrix. Hence, better mechanical properties can be expected in the case of nanocomposite which meant the effect of matrix-to-reinforcement particle size ratio on the present nanocomposites was almost nil.

5.5. Mechanical behavior of nanocomposites

5.5.1. Compressive stress-strain curves of nanocomposites

The mechanical behavior in terms of simple uniaxial compressive stress-strain curves at room temperature (303 K) of AA 6061-x wt.% TiO$_2$ (x=0, 4, 8 and 12 wt.%) nanocomposites is shown in Figure 18. From Figure 18, it was observed that the compressive stress flow curve was started to increase drastically when the amount of reinforcement of TiO$_2$ ceramic particles increases in the nanocrystallite matrix. Maximum ultimate compressive strength of 442 MPa was obtained in AA 6061-12 wt.% TiO$_2$ nanocomposite which was 1.6 times higher than AA 6061 nanocrystallite alloy. The observed ultimate compressive strengths were 275, 295, 365 and 442 MPa for 0, 4, 8 and 12 wt.% TiO$_2$-reinforced nanocomposites, respectively. The corresponding decreased compressive strains were 0.076, 0.065, 0.052 and 0.039 for 0, 4, 8 and 12 wt.% TiO$_2$-reinforced nanocomposites, respectively. The increase in value of compressive strength and decrease in strain with the function of nano-TiO$_2$ particles were attributed to

<table>
<thead>
<tr>
<th>Composition</th>
<th>Vickers hardness, H$_{1.0}$ (MPa) (function of sintering temperature)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>400°C</td>
</tr>
<tr>
<td>6061 Al alloy</td>
<td>488.14±35.6</td>
</tr>
<tr>
<td>6061 Al alloy+2% TiO$_2$</td>
<td>506.24±42.3</td>
</tr>
<tr>
<td>6061 Al alloy+4% TiO$_2$</td>
<td>533.22±53.9</td>
</tr>
<tr>
<td>6061 Al alloy+6% TiO$_2$</td>
<td>570.15±52.4</td>
</tr>
<tr>
<td>6061 Al alloy+8% TiO$_2$</td>
<td>608.73±62.4</td>
</tr>
<tr>
<td>6061 Al alloy+10% TiO$_2$</td>
<td>621.35±42.8</td>
</tr>
<tr>
<td>6061 Al alloy+12% TiO$_2$</td>
<td>628.90±63.9</td>
</tr>
</tbody>
</table>

Table 3. Vickers hardness as a function of sintering temperature.
Figure 17. SEM/BSEI of sintered nanocomposite AA 6061_\text{wt.\%} TiO\textsubscript{2} at 550°C. Left side of (a), (c) and (e) shows 2, 6 and 12 wt.% TiO\textsubscript{2} nanocomposite. Right side of (b), (d) and (f) shows magnified view of corresponding sintered preforms. Note: double arrow represents the distribution of TiO\textsubscript{2} particles.
crystallite refinement in the matrix, uniform distribution and embedding of reinforcements over the matrix. Due to this, effective load transfer between matrix and reinforcement had occurred.

5.5.2. Finite element simulation of compressive stress-strain curves of nanocomposites

Finite element simulations were also carried out using simple uniaxial compressive stress-strain data. In the simulation, 3D solid model of 5 mm×5 mm×10 mm as per ASTM standard specimen was used; one end of model was set as fixed support, and the other end was used to give compressive load. Before simulations, the material properties such as density, Young’s modulus value, Poisson’s ratio and ultimate compressive strength were given as input in ANSYS workbench software. Figure 18 in dashed line shows the finite element results. Excellent agreement between experimental results and finite element results was observed. Figure 19a and b shows the stress distribution at ultimate compressive strength of AA 6061 and AA 6061-12 wt.% TiO₂ nanocomposites, respectively. It can be observed from Figure 19 that the stress concentration at the bottom of developed/simulated model was more in AA 6061-12 wt.% TiO₂ nanocomposite when compared to AA 6061 nanocrystallite alloy. This was attributed to more crystallite refinement, uniform distribution of nano TiO₂ particle and more amount of embedding of TiO₂ ceramic particles over the matrix.
5.5.3. Compressive stress-strain curves of trimodeled nanocomposites

It was observed from Figure 18 that the manufactured nanocomposites exhibited highest compressive strength. However, it showed poor in toughness and ductility. Therefore, in order to improve toughness and ductility by sacrificing the strength slightly, trimodel-based nanocomposites were successfully fabricated. These trimodel nanocomposites were consisting of nanocrystallite matrix (after 40-h MA), nano TiO$_2$ particles (~119 nm) and coarse crystallite matrix. It was expected that the addition of coarse crystallite matrix in the nanostructured materials would enhance the toughness and ductility. As an example, AA 6061-12 wt.% TiO$_2$ nanocomposites (after 40-h MA) were taken here for investigation. Figure 20 shows the compressive stress-strain curves of AA 6061-12 wt.% TiO$_2$-y wt.% CG nanocomposites (y=0, 5, 10, 15, 20, 25 and 30) of both experiment (Figure 20a) and finite element (Figure 20b). Inspection of AA 6061-12 wt.% TiO$_2$ nanocomposite curves showed that there was a poor strain (poor in ductility) of around 0.039 mm/mm only; however, it exhibited an ultimate compressive strength of 442 MPa. The poor value of strain (ductility) for nanostructured alloy/composites via MA was attributed to MA processing which causes residual moisture during cold consolidation led to pores after sintering [50], segregation of impurities created by milling media (i.e., balls and vial material) [51] and the dispersoid-matrix interface de-cohesion [52]. The measured ultimate compressive strength for 0, 5, 10, 15, 20, 25 and 30% CG nanocomposites were 442, 449, 454, 474, 461, 438 and 427 MPa, respectively. These results revealed that the ultimate compressive strength was started to increase up to 15 wt.% CG matrix in AA 6061-12 wt.% TiO$_2$ nanocomposites, and then, it started to decrease from 20 to 30 wt.% CG matrix in the nanocomposites. Indeed, as the CG matrix increases in the nanocomposites, it tries to accommodate the dislocation mobility. Further, the CG tries to arrest the crack propagation that usually occurs in nanocomposites as it is subjected either tensile force or compressive force. Around 7% of increased ultimate compressive strength was observed in AA 6061-12 wt.% TiO$_2$-15 wt.% CG nanocomposites when compared to AA 6061-12 wt.% TiO$_2$ nanocomposite. The corresponding compressive strain was 0.039, 0.050, 0.057 and 0.071 mm/mm for 0, 5, 10 and 15% CG nanocomposites, respectively. This was attributed to enhanced densification by increasing soft phase and uniform distribution of CG matrix in nanocomposite structures. However, the observed ultimate compressive strength started to decrease when the CG matrix addition was beyond 15%. This was expected to the domination of softer CG matrix and coalescence of CG particles which might have decreased the effective load transfer rate. In contrast, the ductility was improved in a better manner as the large number of CG matrix phase in the nanocomposites might have effectively delayed the crack initiation in nanocomposites during deformation. The compressive ductility for 30% CG trimodel nanocomposite was around 520-0% CG nanocomposites. To conform the uniform distribution and coalescence of CG matrix in nanocomposite, the microstructures of AA 6061-12 TiO$_2$-y wt.% CG (y=0, 10, 20 and 30) nanocomposites were taken and the same is shown in Figure 20a–d. From the microstructures, two different regions, namely, gray regions and white regions were seen. The gray region characterizes ultra-fine grain (UFG) matrix phase embedded with nano-TiO$_2$ ceramic particles, whereas the white region characterizes CG matrix phase. From Figure 21b–d, it was very clear that a good homogeneous distribution of CG matrix phase was observed in UFG matrix. Further, it can be observed that as the percentage of CG matrix phase increases, the size of CG domain also increases. Due to this, the dislocations move a significant distance without crystallite boundary interruption when trimodel material is deformed [53, 54]. Therefore, it was expected to
Figure 19. Stress distribution at ultimate compressive strength of simulated model of: (a) AA 6061 nanocrystalline alloy, (b) AA 6061-12 wt.% TiO$_2$ nanocomposite.
Figure 20. Compressive stress-strain curves of AA 6061-12 wt.% TiO$_2$ – y wt.% CG nanocomposites (y=0, 5, 10, 15, 20, 25 and 30): (a) experimental results and (b) finite element results.

Figure 21. Trimodel microstructures of as-sintered AA 6061-12 wt.% TiO$_2$ composites containing x wt.% CG matrix: (a) x=0%, (b) x=10%, (c) x=20% and (d) x=30%. The gray regions represent UFG matrix reinforced with nano Titania, the bright regions represent CG matrix.
have improvement in ductility of nanocomposite material by incorporating CG matrix. The size of CG matrix phase was not increased up to 15% CG-blended nanocomposites. The measured average CG size was less than 45 μm up to 15% CG-blended composites due to non-coalescence of individual CG matrix particles. Conversely, the average size of CG matrix phase started to increase when CG matrix content increases beyond 15% (Figure 21c and d).

From Figure 20d, AA 6061-12 wt. TiO2-30% CG nanocomposite is clearly showing a very large size of CG matrix phases due to coalescence of individual CG matrix particles. Witkin et al. [55] have also observed the same behavior while studying the bimodal crystallite size of Al-Mg alloy (0, 15 and 30% CG matrix phase) prepared by cryomilling and blending. Due to this, a good ductility was observed by slight decrement of compressive strength.

6. Summary and conclusions

A new nanocomposite of AA 6061 Al alloy reinforced with different weight percentage of titania particles was developed and was successfully synthesized and investigated for its mechanical behavior. It was found that the matrix powder particle size, shape and its morphology, reinforcement particle size and its distribution on the matrix, amount of reinforcement, crystallite size of the matrix and type of microstructures (bimodal and/or trimodal) were reflected and influenced the performance of the fabricated light weight-based nanocomposite which is suitable for automotive and structural applications.

6.1. Powder surface morphology

The powder morphology of nanocomposite powders with the function of reinforcement was investigated. It was experimentally found that the incorporation of hard ceramic reinforcements in the nanocomposite powders exhibited drastic changes on the morphological characteristics of MMCs. The produced nanocomposite powders morphology showed that all powder particles regardless of the reinforcement were almost in spherical-shaped and equiaxed particles which indicated the attainment of steady-state condition during 40-h MA. On the other hand, the irregular flake-like morphology of as-received soft matrix powder particles was eliminated after 40-h MA and changed to a shape near the equiaxed. Further, nano-sized TiO$_2$ particles are completely embedded in soft matrix of nanocomposite powder after 40 MA. There is no way of forming of reinforcement clustering in nanocomposite powder via MA during the time taken (40 h) for the present investigation.

6.2. Structural evaluation

The structural characteristics of ball-milled mechanically alloyed powders were investigated and reported. These results confirmed the occurrence of structural refinement in nanocomposites with the function of reinforcement through mechanical alloying, and hence, decreased crystallite size was observed. The MA time selected for this study produced the crystallite size of around 46 and 65 nm for 12 wt.% reinforced particulate nanocomposite and unreinforced nanocrystallite powders, respectively. The nanocrystalline nature of the matrix powder particle and aggregation of nanocomposite powder particle was confirmed by TEM analysis.
6.3. Mechanical strength and sintering behavior

Nanostructured AA 6061 Al alloy reinforced with different weight percentage of TiO$_2$ particles processed via MA followed by cold consolidation and sintering yielded maximum strength (hardness) of around 1.126 GPa of matrix grain size of 200–300 nm (UFG) embedded with nano-sized Titania (~119 nm). This was attributed to structural refinement contributed from various strengthening mechanisms such as grain size, dislocation, solid solution and dispersion strengthening. Here, the matrix to reinforcement particle size ratio of nanocomposite was greater than unity and even very higher value. However, excellent behavior in terms of densification, sinterability and Vickers hardness was obtained. These results indicated that the effect of matrix to reinforcement particle size ratio was insignificant in the case of nanocomposite because there was no possibility of reinforcement clusters while manufacturing of MMCs via MA.

6.4. Compressive stress-strain behavior

The compressive stress-strain curves of nanocomposites were started to increase drastically with the function of reinforcement which indicated the enhancement of compressive strength while it exhibited the decreased value of ductility with the function of reinforcement. These results were attributed to more crystallite refinement and complete embedding of titania ceramic particles in the matrix. A trimodel grain size distribution for enhancing the ductility and toughness of high strength AA 6061–TiO$_2$ nanocomposites was fabricated successfully and the uniaxial compressive deformation behavior through simple compression test at room temperature was studied, analyzed and reported. These results explained that the addition of coarse crystallite powder particles in the nanostructured materials would enhance the ductility, delay the plastic instability and enhance toughness. In other words, the addition of CG matrix phase decelerates the micro-crack nucleation cum proliferation in nanostructured phase during deformation. Therefore, the ductility in nanocomposite materials can be improved which would be suitable for manufacturing of automotive and structural components. Here, the 30% CG-incorporated nanocomposite exhibited improved ductility of around 500% more than that of 0% CG incorporated nanocomposite.

Author details

S. Sivasankaran* and Abdulaziz S. Alaboodi

*Address all correspondence to: sivasankarangs1979@gmail.com

Department of Mechanical Engineering, College of Engineering, Qassim University, Kingdom of Saudi Arabia

References


[34] Sivasankaran, S., K. Sivaprasad, R. Narayanasamy and P.V. Satyanarayana. (2011) X-ray peak broadening analysis of AA 6061_{100-x} wt.% Al_{2}O_{3} nanocomposite prepared by mechanical alloying, Mater. Charact., 62, 661–672.


