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A Review on Properties, Behaviour and Processing Methods for Al-Nano Al$_2$O$_3$ Composites

Dinesh Kumar Koli[a], Geeta Agnihotri[b], Rajesh Purohit[c]

[a]*Research Scholar, Maulana Azad National Institute of Technology, Bhopal, India-462051*
[b]*Professor, Maulana Azad National Institute of Technology, Bhopal, India-462051*
[c]*Associate Professor, Maulana Azad National Institute of Technology, Bhopal, India-462051*

Abstract

Nanotechnology is spreading vastly in the various demanding fields of engineering and medicines like aerospace, defence, automobiles, electronics, materials, chemistry, energy, environment, information & communication, consumer goods and bio-technology. It created a high impact on development of new generation nano materials with advanced features and wide range of their applications. Reinforcement of submicron or nano-sized particles with aluminium matrix yields superior mechanical and physical properties and changes morphology and interfacial characteristics of nano-composites. A wide range of research has been done on the processing methods and material properties of Al-Al$_2$O$_3$ nano-composites. Recently, ultrasonic assisted casting, nano-sintering, powder metallurgy, high energy ball milling, friction stir process are being applied for the production of Al-Al$_2$O$_3$ nano-composites in which agglomeration of the reinforcing particles causes grain growth resulting changes in the microstructure. Control of the grain size by minimizing agglomeration of nano particles and retaining the enhanced microstructure during these processes has become a challenging task. This area is further looked-for research work to control the microstructures under various processing conditions.

1. Introduction

Nanotechnology refers to the field of engineering and technology which controls matter on an atomic and molecular scale. It deals with structures with at least one dimension in the nanometer range or less than 100 nanometers, about one-thousandth the diameter of a human hair, the scale of the atoms or small molecules. Below 1

* Corresponding author. Tel.: +0-000-000-0000 ; fax: +0-000-000-0000 .
 E-mail address: r_purohit@rediffmail.com

nanometer, the properties of materials become familiar and predictable (the domain of chemistry and atomic physics). Edward et al. (2010); Froes (1998); Seal et al. (2004); Timms, Ponton and Strangwood (2002) shown that nanotechnology is a wide ranging technology which finds applications in many technical disciplines including but not limited to chemistry, biology, physics, material science, electronics, MEMS, self-assembly etc. Nano-structures have the ability to generate new features and perform new functions that are more efficient than and/or cannot be performed by larger structures and machines. Nanotechnology is based upon new approaches like molecular self-assembly with dimensions on the nano scale to investigate whether we can directly control matter on the atomic scale. Seal et al. (2004); Veeresh, Rao and Selvaraj (2011) and Yung-Chang and Sammy (2004) reported that due to the small dimensions of nano-materials, their physical/chemical properties (e.g. stability, hardness, conductivity, reactivity, optical sensitivity, melting point, etc. can be manipulated to improve the overall properties of conventional materials. At nanometer scales, the surface properties start becoming more popular than the bulk material properties, generating unique material attributes and chemical reactions. More essentially, the electronic structure of materials becomes size-dependent as the dimensions enter the nanoscale. Hence, the optical properties, including light absorption and emission behavior, will be altered. The fact that nanoscale features are smaller than the wavelength of visible photons also impacts light scattering, enabling the design of nano-crystalline ceramics that are as transparent as glass. Changes in the bonding at the surface of a nano particle will affect the electronic structure as well, and the implications for the reactivity of the surface can be significant. Materials reduced to the nano scale can show different properties compared to what they exhibit on a macro scale, enabling unique applications. One example is the increase in surface area to volume ratio altering mechanical, thermal and catalytic properties of materials. For example, opaque substances become transparent (copper); stable materials turn combustible (aluminum); insoluble materials become soluble (gold). A material such as gold, which is chemically inert at normal scales, can serve as a potent chemical catalyst at nano scales. Much of the fascination with nanotechnology stems from these quantum and surface phenomena that matter exhibits at the nano scale.

1.1 Nanocomposites

Composite material is a mixture of two or more materials or insoluble in one another, possessing properties which are superior to any of the component materials. Pedro et al. (2009); Seal et. al. (2004); Vencl, Rac and Bobic (2004) defined nanocomposites as a class of materials that contain at least one phase with constituents in the nanometre size range (1 nm = 10^{-9} m). Nanocomposite is a multi-phase material which has nano particles in its composition within its structure, the size will be less than 100 nm. The properties including mechanical, electrical and thermal may differ depending upon the composition of the materials used for the synthesis of the composites. Ozdemir et al. (2008) and Veeresh, Rao and Selvaraj (2011) shown that the structure and the properties of nano-composites are controlled by the type and size of the reinforcement, nature of bonding and processing technique The amount, size and distribution of reinforcing particles in the metal matrix play an important and critical role in enhancing or limiting the overall properties of the composite material. Reinforcing aluminium matrix with much smaller particles, submicron or nano-sized range, is one of the key factor in producing high-performance composites, which yields improved mechanical properties A simple example of a normal composite is the concrete in our houses. It is a composite of cement, sand, and metal rod. These composition changes the overall property of the material used. It becomes so hard that it can withstand tonnes of load equally. The mechanical, electrical, thermal, optical, electrochemical and catalytic properties of the nanocomposite will differ markedly from that of the component materials. Pedro, Satyanarayana and Fernando (2009) reported that certain size effects which govern the property of these materials:

- Less than 5 nm for catalytic activities
- Less than 20 nm for making hard magnetic materials
- Less than 50 nm for refractive index changes
- Less than 100 nm for achieving super magnetism
- Less than 100 nm for Mechanical strengthening or restricting matrix dislocation movement
- Less than 100 nm for Producing toughening

- Less than 100 nm for Modifying hardness and plasticity

Ronald (2010) has explained that nanocomposites differ from conventional composite materials due to the exceptionally high surface to volume ratio of the reinforcing phase or its exceptionally high aspect ratio. The surface area-to-volume ratio, A/V, for a spherical particle is inversely proportional to its radius, so A/V for a nanometre-sized particle will be 1000 times greater than A/V for a micron-sized particle. The large A/V ratios and resulting large pore volumes for porous hollow nanostructures make them particularly attractive for multifunctional delivery of drugs and bio-molecules. The reinforcing material can be made up of particles (e.g. minerals), sheets (e.g. exfoliated clay stacks) or fibres (e.g. carbon nanotubes). The area of the interface between the matrix and reinforcement phase(s) is typically an order of magnitude greater than for conventional composite materials. The matrix material properties are significantly affected in the vicinity of the reinforcement. This large amount of reinforcement surface area means that a relatively small amount of nanoscale reinforcement can have an observable effect on the macro scale properties of the composite. For example, adding carbon nanotubes improves the electrical and thermal conductivity. Other kinds of nano particulates may result in enhanced optical properties, dielectric properties, heat resistance or mechanical properties such as stiffness, strength and resistance to wear and damage. In general, the nano reinforcement is dispersed into the matrix during processing. The percentage by weight is of the order of 0.5% to 5% due to the low filler percolation threshold, especially for the most commonly used non-spherical, high aspect ratio fillers (e.g. nanometre-thin platelets, such as clays, or nanometre-diameter cylinders, such as carbon nanotubes).

1.2 Types of Nanocomposites

a) Ceramic Matrix Nanocomposites (CMNC)
b) Metal Matrix Nanocomposites (MMNC)
c) Polymer Matrix Nanocomposites (PMNC)

a) CERAMIC MATRIX NANOCOMPOSITES (CMNC):- In this group of composites the main part of the volume is occupied by a ceramic, i.e. a chemical compound from the group of oxides, nitrides, borides, silicides etc. In most cases, ceramic-matrix nanocomposites encompass a metal as the second component. Ideally both components, the metallic one and the ceramic one, are finely dispersed in each other in order to elicit the particular nanoscopic properties. Nanocomposites improve their optical, electrical and magnetic properties as well as tribological, corrosion-resistance and other protective properties. Ceramic Matrix Nanocomposites include Al_2O_3/SiO_2, SiO_2/Ni, Al_2O_3/TiO_2, Al_2O_3/SiC, Al_2O_3/CNT etc.

b) METAL MATRIX NANOCOMPOSITES (MMNC):- Metal matrix nanocomposites (MMNC) refer to materials consisting of a ductile metal or alloy matrix in which some nanosized reinforcement material is implanted. These materials combine metal and ceramic features, i.e., ductility and toughness with high strength and modulus. Thus, metal matrix nanocomposites are suitable for production of materials with high strength in shear/compression processes and high service temperature capabilities. They show an extraordinary potential for application in many areas, such as aerospace, automotive industries and other Metal Matrix Nanocomposites include Al/Al_2O_3, Al/SiC, $Fe-Cr/ Al_2O_3$, Ni/ Al_2O_3, Co/Cr, Fe/MgO, Al/CNT, Mg/CNT etc.

c) POLYMER MATRIX NANOCOMPOSITES (PMNC):- Adding nano particles to a polymer matrix can enhance its performance, often in very dramatic degree, by simply capitalizing on the nature and properties of the nanoscale filler. This strategy is particularly effective in yielding high performance composites, when good dispersion of the filler is achieved and the properties of the nanoscale filler are substantially different or better than those of the matrix, e.g. reinforcing a polymer matrix by much stiffer nanoparticles of ceramics, clays, or carbon nanotubes. Polymer Matrix Nanocomposites include thermoplastic/thermoset polymer/layered silicates, polyester/TiO_2, polymer/CNT, polymer/layered double hydroxides.

1.3 Advantages of Nanocomposites

Nano-composite materials have emerged as suitable alternatives to overcome limitations of micro-composites

and monolithics, while posing preparation challenges related to the control of elemental composition and stoichiometry in the nanocluster phase. The following are the advantages of nano-composites over conventional materials:

1. Greater tensile and flexural strength as compared to matrix material.
2. Reduced weight for the same performance.
3. Increased dimensional stability.
4. High modulus of elasticity and wear resistance.
5. High thermal stability.
6. Improved gas barrier properties for the same film thickness.
7. Flame retardant properties.
8. High temperature creep resistance.
9. Improved specific strength and stiffness.
10. Improved fracture toughness and thermal shock resistance.
11. Higher electrical conductivity.
12. Higher chemical resistance.

1.4 Applications of Nanocomposites

Edward et al. (2010); Froes (1994); Hesabi, Simchi and Reihani (2006); Mahboob, Sajjadi and Zebarjad (2008); Woo and Lee, (2007); Ronald (2010) have stated the applications of nanocomposites in various fields like aerospace, defence, automobiles medicine, electronics, materials, marine, industrial and construction markets etc. The other applications are:

1. Thin-film capacitors for computer chips.
2. Solid polymer electrolytes for batteries.
3. Automotive engine parts and fuel tanks.
4. Impellers and blades.
5. Oxygen and gas barriers.
6. Food packaging.
7. Drug delivery systems.
8. Anti-corrosion barrier coatings.
9. UV protection gels.
10. Lubricants and scratch free paints.
11. New fire retardant materials.
12. New scratch/abrasion resistant materials.
13. Superior strength fibers and films.

1.5 Al/Al$_2$O$_3$ Nanocomposites

Tousi et al. (2009) shown in their study that due to light weight and high specific strength, particulate reinforced aluminium composites are attractive structural materials for various domains such as automotive and aerospace applications. An optimum combination of high strength and ductility gives Aluminium metal matrix nanocomposites (AMNC) a wide range of advanced applications. Mazen and Ahmed (1998); Mula et al. (2009) made comparison of pure Al with 2.0 vol% nano-Al$_2$O$_3$ additions and found that improvement in yield strength of around 66%, hardness of around 50% and tensile strength of around 80%. Dominique et al. (2010) reported in their experiment of ultrasonic assisted casting to disperse 2.0 wt% nano-Al$_2$O$_3$ (10 nm) in aluminium matrix. In Comparison to pure Al cast by the same method, composite hardness was increased by 92% and the yield strength by 56%. Hafeez and Senthil (2010) resulted that because of low density, low melting point, high specific strength and thermal conductivity of aluminium, a wide variety of reinforcement particulates such as Al$_2$O$_3$, SiC, B$_4$C, AlN, Si$_3$N$_4$ TiC, TiO$_2$, TiB$_2$ and graphite have been reinforced into it. Among these particulates, Al$_2$O$_3$, SiC, B$_4$C, TiB$_2$ additions improved the wear behaviour of aluminium matrix composites.

1.1. Powder Metallurgy

The basic process of powder metallurgy consists of three major stages. First, the primary material is physically powdered into many small individual particles. Then, the different powders of metals and /or ceramics are mixed in required proportions. Ball milling may be performed to achieve mechanical alloying of powder mixtures for better mechanical properties. Next, the powder is injected into a mold or die and compacted on a press to produce a weakly cohesive structure close to the dimensions of the object ultimately to be manufactured. Finally, the product is formed by applying high temperature, pressure, long setting times, or any combination thereof. After, secondary operations like extrusion, heat treatment or machining etc. may be done.

Mazen and Ahmed (1998) mixed pre-weighed pure alumina powder (Al_2O_3) with pure aluminium (Al) powder through the use of a mechanical mixer and four different compositions, Al-0wt%Al_2O_3, Al-2.5wt%Al_2O_3, Al-5wt%Al_2O_3, & Al-10wt%Al_2O_3 were prepared. The Al-Al_2O_3 powder mixture was then hot pressed at 723 K for 4 h using the hot-pressing setup shown in Fig.1, using a compaction pressure of 74 MPa on the 24 mm diameter billets. The hot-pressed billets were then hot extruded and the extruded rods were used as specimens for different tests. SEM revealed the presence of porosity and particle-rich areas in the matrix as well as debonding of some alumina particles under fracto-graphic examination due to weak bond strength and large difference between the melting points of Al and Al_2O_3. In liquid phase processing, wettability is achieved only at high processing temperatures (>1173 K).
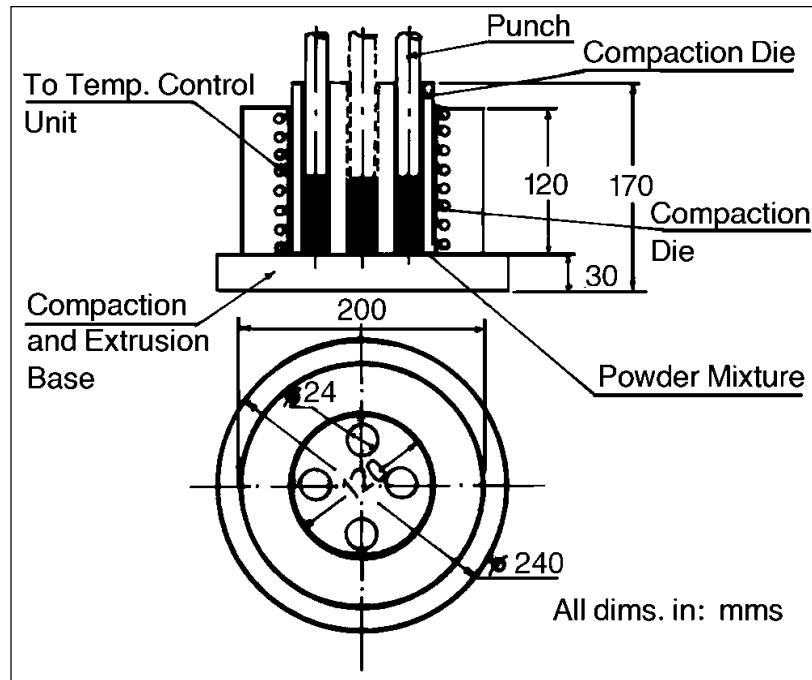

Fig.1. Hot pressing and extrusion setup

Yung-Chang and Sammy (2004) took commercial pure aluminium powder containing 38 ppm Cu, 20 ppm Si, 38 ppm Mg, 18 ppm Mn, and 1200 ppm Fe. The mean particle size of this gas-atomized powder was about 28μm. Nanometric spherical Al_2O_3 powder with a purity of at least 98.5% was used as the reinforcement. The mean particle size was about 50 nm. The aluminium powder was mixed with different volume fraction (1–7 vol.%) of Al_2O_3 powder in pure ethanol slurry. Mixed powders were dried at 150°C and then compacted by CIP. In order to compare the mechanical properties of the nano-metric particle-reinforced aluminium composite with the

micrometric particle-reinforced aluminium composite, a 10 vol.% SiC_p (13 μm)/Al composite was fabricated following the same route. All compacted billets were sintered in vacuum at 620°C for 2 h. Finally, the sintered compacts were extruded at 420°C with a reduction ratio of approximately 36:1, to form bars of 15mm in diameter. All specimens were subjected to an anneal heat treatment at 350°C for 2 h. The result showed that the nanometric particulates improve the mechanical properties in the monolithic system. The major strength mechanism was the Orowan strengthening by these particulates. However, as the particulate volume fraction increased, the extent of agglomerations of the particulates increased, when it exceeded 4 vol.%, the agglomerations reduce the amount of 'effective' nano-particulates available, and the particle strengthening effect diminishes.

2. Processsing Methods of Al/Al$_2$O$_3$ Nanocomposites

Metal matrix nanocomposites (MMNCs) are manufactured using different techniques. These techniques could be classified as (a) liquid-state processes: stir casting, squeeze casting, ultrasonic-assisted casting, vacuum infiltration , pressureless infiltration, and dispersion methods, (b) solid state processes: powder metallurgy (PM) techniques with variations in the processing steps e.g. hot iso-static pressing, cold iso-static pressing, hot die pressing, dynamic compaction , and (c) liquid-solid processing: compo-casting, semisolid forming. The limitations of the first and third groups arise from difficulties in mixing the two phases thoroughly, difficult determination of critical temperature for infiltration, problems due to fluidity and/or wettability at matrix-reinforcement interface, as well as harmful reactions at the interface.

2.1.1 Mechanical Milling

Mechanical milling is a process involving repeated deformation, welding and fracture. Many parameters such as miller type, ball to powder weight ratio, characteristics of the balls and their speed, milling atmosphere and temperature, and process control agent influence the stages of milling. Upon milling of composite powders, the volume fraction, the particle size and the type of the reinforcement also affect the process.
Razavi et al. (2006) produced aluminium powder by nitrogen gas atomization process. The particle size and morphology of Al powder were determined by a laser particle size analyzer and a SEM respectively and found that the particles are almost spherical with the mean diameter of 48μm. Two grades of α-Al$_2$O$_3$ powders with average particle size of 35 nm (n-Al$_2$O$_3$) and 1μm Al$_2$O$_3$) were used. Stearic acid powder was used as the process control agent (PCA). The Al powder was blended with 5 vol% Al$_2$O$_3$ and 1.5 wt% PCA in a Turbula T2C mixer for 20 min. The powder mixtures were then mechanically milled in a planetary ball mill with a hardened steel vessel under a high purity argon atmosphere up to 24 h. The rotational speeds of 250 rpm and ball to powder weight ratio of 10:1 were employed. In different stages of MA, small amount of the milled powders were collected for testing. Aluminum powder without alumina addition was also processed at the same condition. It was found that the milling stages include plastic deformation, micro-welding, and particle fragmentation. When hard alumina particles are added to aluminum powder, the fracture occurs earlier, and thus the steady-state condition, i.e. formation of equi-axed particles, is achieved after shorter milling time. The bulk density of composite powders was found to be higher than that of unreinforced Al. Meanwhile, it was shown that the mechanical milling stages occur earlier in Al–Al$_2$O$_3$ micro-composite compared to the nano-composite.

Hesabi et al. (2007) utilized nitrogen gas atomized Al powder with mean particle diameter of 49 μm, Al$_2$O$_3$ nano-particles with average size of 35 nm, and stearic acid were used as the starting materials. Al–5.0 vol.% Al$_2$O$_3$ composite powder was prepared by mechanical blending of the starting powders in a Turbula T2C mixer for 20 min. Steel balls with diameter of 10 mm and the ball to powder charge ratio of 1:2 (wt.%) were used. In order to study the effect of mechanical milling on the compressibility behavior of the composite powder, the Al–5vol.% Al$_2$O$_3$ mixture was milled for 72 hrs in a planetary ball mill under a high purity argon atmosphere. 1.5 wt.% stearic acid was used as the milling process control agent (PCA). The rotational speed of 250 rpm and the ball to powder charge ratio (wt.%) of 10:1 were employed. Monolithic aluminum powder was also processed at the same condition to study the effect of the hard nano particles on the compressibility. The compressibility of Al and Al–5 vol.% n-Al$_2$O$_3$ powders during uniaxial compaction was investigated. The compressibility of the blended and milled aluminium/nanometric alumina particles exhibits the same features as the typical metal powder compaction. The consolidation is mainly

generated by two phenomena: particle rearrangement (Stage I) and plastic deformation (Stage II). The addition of hard nanoparticles to aluminium powder by mixing improves the densification capacity at Stage I due to disintegration of the clusters and agglomerates under the applied load and filling the voids between the matrix particles. The plastic deformation capacity of the milled composite powder was found to be less than that of the blended powder, showing the effect of distributed nanoparticles on the work-hardening rate and the hardness of the aluminium matrix.

Woo and Lee (2007) used pure Al, Mg, Cu, Si and SiO_2 powder (average particle size 70, 30, 20, 50 and 40μm, respectively) in their experiments. The compositions of the powder mixture were Al–0.4 wt.% Mg–14 wt.% SiO2 and Al–3 wt.% Cu–3 wt.% Si–9 wt.% SiO_2 powder, respectively. To prevent powder oxidation, the powders were sealed in stainless steel vial under an argon atmosphere during ball milling. The weight ratio of ball to powder was 4:1. Milling of the mixed powders was conducted for 1–8 h using an SPEX 8000 Mixer/Mill. The characterization of the microstructure for the as-milled powder was performed using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Differential thermal analysis (DTA) was conducted to determine the temperatures at which the displacement reactions between Al and SiO_2 took place during heating from ambient temperature to 800°C with heating rate 10°C/min. The green sample was sintered at 650°C and 750°C for 2 h in a tube furnace under vacuum. Nano-sized SiO_2 particles embedded into the Al alloy matrix was successfully fabricated by HEMM. Nano- and/or subsive-size Al_2O_3 particles were homogeneously distributed in the Al matrix. High energy ball milling can decrease the temperature of the in situ displacement reaction and increase the sintering rate. The flexural strength of the sintered specimen (1.2 GPa) using Al–0.4% Mg–14% SiO_2 powder milled for 8 h was about four times that of as the sintered specimen (300MPa) using as-mixed powder.

Durai et al. (2007) used aluminum powders (99.7% purity, <20 μm size), ZnO and CuO powders (99.5% purity, <10μm size). Powders mixtures have been subjected to a high-energy ball milling using a Fritsch Pulverisette-5 mill with tungsten balls and vials. Tungsten balls with a diameter of 10mm have been used to mill the powder upto 60 h using a ball to powder weight ratio of 10:1. Milling has been carried out at 300 rpm using a toluene medium in order to avoid oxidation or sticking of powders on the wall of the vial. Mechanically milled powders have been cold pressed under a pressure of 650 MPa to form green compacts of 10mm diameter and 4mm length, followed by heating at a given temperature to initiate the reaction in argon atmosphere. The crystallite size in the milled and sintered samples has been determined using the X-ray peak broadening techniques. The results cleared that an improvement in wear resistance is a strong function of Al_2O_3 content and size of Al_2O_3 particles as well as crystallite size of the matrix alloy. Mechanical milling causes a uniform distribution of the particle with small inter-particle distance resulting in an improved wear resistance. The inter-particle distance is expected to decrease with a decrease in reinforcement size. Hardness of the milled composites is higher than the unmilled composites, because the alumina particles of the milled composite are finer and more homogeneously distributed than those in the unmilled composites. This is attributed to the finer crystallite structure obtained after milling. The wear rate increases linearly with the applied load irrespective of the material and decreases with the increasing sliding distance.

Ismail et al. (2008) manufactured the chemical composition of aluminium alloy powder EN AW-2017 by gas atomization process is 3.9%Cu, 0.6%Mn, 0.7%Mg, bal. Al (wt.%). Gas atomized aluminum alloy powders with a mesh size of ~100μm was supplied as the starting matrix material. In order to investigate the influences of reinforcement type, size and volume fraction on producing composite particles, the matrix powder was mixed with volume fractions of 5, and 15% commercially available SiC and Al_2O_3 powders. Al_2O_3 and SiC particles were used as reinforcement with a particle size of −22 + 5 and −55+15μm, respectively. Pre-mixed powders were milled by a laboratory scale high energy ball milling unit. The high-energy milling was performed in argon atmosphere with various rotation speeds (ranging from 600 to 800 rpm) in a stainless steel chamber using stainless steel balls (100Cr6, 4.7mmin diameter ball size) with a powder to ball weight ratio of 1:10. The milling time was varied from 10 min to 8h in case of Al_2O_3 and from 10 min to 6h in case of SiC to investigate its influence on the milled products. HEM process was conducted at room temperature for both types of composite blends. The milled composite powders were taken out at regular intervals of 10 min, 0.5, 1, 1.5, 2, 4, 6 and 8h for microstructural analysis. In this work, Al_2O_{3p} and S_iC_p reinforced ultra fine-grained matrix (3.9Cu, 0.6Mn, 0.7Mg, bal. Al, wt.%) base composites were produced by HEM process. The milling time was varied from 10 min to 6 or 8 h, respectively. At the early stages of milling, i.e., 10–30 min, SiC and Al_2O_3 reinforcement particles are observed rarely inside the

deformed matrix powder. However, a change in the morphology of matrix particles from spherical shape to flake-like was noticeable. In addition, the cellular structure of $CuAl_2$ inter-metallic particles in the matrix powder partially deforms and elongates along the deformation axis. After longer durations of milling a homogenous distribution of reinforcement in the composite powder with smaller particle size was achieved. However, the reinforcing particle size distribution for both SiC and Al_2O_3 particulates showed no significant reduction even after the longest periods of milling. HEM process decreased the crystallite size of the aluminium matrix to about 45 nm. No matter how small (fine Al_2O_{3p}) or how much volume fraction were being utilized (5 or 15 vol.%), no significant effect on further decrease in crystallite size was detected.

H. Mahboob et al. (2008) used commercial aluminium powders with particle size smaller than 63 μm and nano-sized α-alumina powder with %99.5 purity and average size of about 27-43 nm have been provided. Fig. 2. (a) and (b) show micrographs of the α-alumina and aluminum powders taken by TEM and SEM, respectively. It was found that the- alumina particles are almost spherical and aluminium particles are irregular in shape.
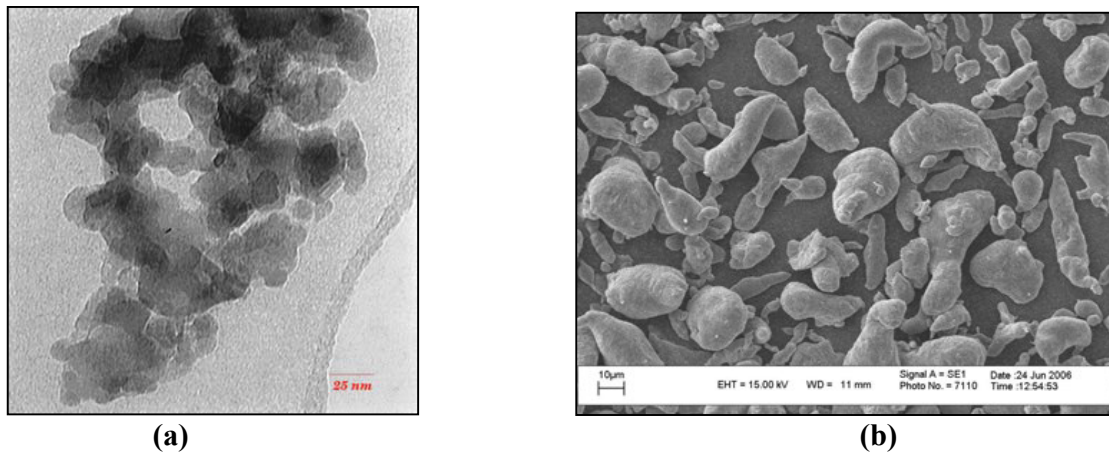

(a)   (b)

Fig.2. Initial used powders: (a) α-alumina, (b) aluminium

High energy planetary ball mill with stainless steel balls with different diameters (8.5-10 mm) were employed. Rotary velocity of the mill and ball to powder weight ratio were 250 rpm and 15, respectively. The aluminum and 5wt% alumina nano powders were milled at different times, i.e., 0, 4, 8 and 12 hours. Process control agent was also used to prevent powders from severe cold welding and contamination. Diameter and height of the cylindrical samples produced using powder metallurgy method were 10 and 15mm, respectively. Samples were fabricated under 420MPa pressure and after five minutes, pressure was removed. For sintering, the compressed powders were kept at 624-626ºC for 45 min under argon gas atmosphere and then the samples were furnace cooled. The results of the research show that Al- Al_2O_3 nano composite mechanical properties concern to milling time. The results in this research show that the microstructure becomes more uniform and fine as a result of milling. Strength and hardness were increased by increasing of milling time but the elongation was almost constant. Al-5%wt. Al_2O_3 composite strength was appreciably increased by 12 hours milling, due to the uniform distribution of nano-size alumina particles. Because of nano size reinforcements, alumina particles inhibit elongation to reduce.

Razavi et al. (2009) utilized commercial purity Al powder as a monolithic system and a mixture of Al–20wt.% alumina powder (Martinswerk, MR70, d50: 0.5–0.8 μm)were separately milled in a P5 planetary mill for various periods of time up to 25 h. The ball to powder ratio was approximately 15:1 and the mill speed was maintained at 250 RPM. 3 wt.% of stearic acid as process control agent (PCA) was added to retard excessive welding. The milling atmosphere was Ar which was purged into the cups before milling. Product sampling was performed in the glove box in the Ar atmosphere to prevent oxidation. The powders after milling were examined using a SEM operating at a voltage of 30 kV. It was found that the addition of alumina powder has a great influence on the morphological

characteristics of MMCs, and also worked to decrease the time taken to reach a steady state. This is based on the observed correlation between green and tap density vs. milling time, explained by the morphological and microstructural evolution of the powder particles. The distribution of the alumina particles in the Al matrix reaches a full homogeneity after steady state. The effect of the nanostructured Al matrix, the presence of submicron alumina particles and nanometeric particles caused by decomposition of PCA increase the hardness of powder up to 180 HV.

Tavoosi et al. (2009) taken a mixture of commercial aluminum (99.7% purity and particle size of 50-70μm) and 15.8 wt% ZnO powders (99.9% purity and particle size of 250 nm) was milled in planetary ball mill in order to produce Al–13.8 wt%Zn/5 vol%Al_2O_3 nanocomposite. The MA was executed in a planetary ball mill with a rotation speed of 600 rpm and a ball-powder mass ratio of 15:1 for predetermined hours without interruption. For comparison reasons amixture of aluminum and 13.8 wt% zinc powders (99.7% purity and particle size of 10–20μm) was also milled with a planetary ball mill at the same conditions. The milled powders were consolidated in a uniaxial die at 400 and 500°C under a constant pressure of 400 MPa. Previous to the HP sintering procedure, the powders were cold pressed under 200 MPa pressure into 10mm diameter discs. The duration of hot pressing was 20min. In order to avoid pores formation, the pressure on specimen was released after cooling down to 300°C. It was found after 60 h milling and hot pressing at 500°C under 400 MPa pressure. That the relative density of the hot pressed samples increased (from 95% to 99.6%) as the temperature increased (from 400-500°C). Al crystallite size for both samples remained constant after annealing at 500°C for 20 min. The hardness value for Al–13.8 wt%Zn/5 vol% Al_2O_3 nanocomposite (relative density about 99.6% and crystallite size about 40 nm) was about 180 HV and for Al–13.8 wt%Zn (relative density 99.8% and crystallite size of about 40 nm) was about 150 HV. The variation of hardness vs. annealing time at different temperatures showed that the produced nanocomposite had a good thermal stability at temperatures below 400°C.

Dominique et al. (2010) used three different sizes of Al_2O_3 powders. Spherical Al_2O_3 of 4 nm nominal size was bought from Aldrich. From observations with a FE-SEM Hitachi S-4700, the average particle size was measured to be 25 nm. Spherical Al_2O_3 of 80 nm average particle size was produced by combustion synthesis. Al powder was first milled alone for 5 h under argon atmosphere in a high energy Spex 8000 mill with a rpm of 1200. 2.0 wt% stearic acid was added as a process control agent to avoid excessive sticking and agglomeration of the aluminum. Tungsten carbide balls (11mm) and container with a 10:1 ball-to powder ratio were used. Mixtures of Al_2O_3/Al with concentration varying from 1 to 10 vol% were then milled for each type of alumina powder using the same procedures. The milled Al mixture with no Al_2O_3 addition and the 10 vol% Al_2O_3 mixtures were cold pressed at 500 MPa and then hot pressed in an uniaxial press at a pressure of 350 MPa and at a temperature of 450°C for 1 h under vacuum in order to obtain sintered cylinders of 2.5 cm in diameter and around 0.6cm height. It was found that the Al_2O_3 composites resulting from milling display a uniform dispersion of the second phase with few agglomerates of around 1 micron in size. The Al_2O_3/Al nanocomposite powders hardness is near five times higher than pure unmilled Al. A decrease in the Al_2O_3 particle size from 400 to 4 nm has increased the nanocomposite powder hardness of 11%. Presence of Al_2O_3 also affects the grain refinement occurring during milling. Compression tests performed on the hot pressed compacts have shown similar trends with a final yield stress and compression strength of 661 and 723 MPa respectively for the 10 vol% Al_2O_3 (4 nm) composite. The observed strengthening is associated to grain refinement and dispersoid formation from mechanical milling together with in situ matrix strengthening from the addition of the n- Al_2O_3.

Tabandeh et al. (2010) used air atomized commercial pure aluminium powder with a mean particle size of 45 μm and spherical pure alpha-Al_2O_3 powders with average particle sizes of 35 nm and 0.3 μm. The P/M process was used to produce the composite materials. At first, the nanometric Al_2O_3 powder was de-agglomerated by ultrasonic agitation in ethanol slurry for 1h. Then, the resultant slurry and the sub-micrometric Al_2O_3 powder were poured into an attrition mill container. After 1h attrition milling, the aluminium powders were added to the milled materials, in which hardened steel balls with a diameter of 5 mm were used as abrasive media. The rotational speed of 480 rpm and the ball-to-powder ratio (BPR) of 15:1 (wt. %) were conducted with 8 h milling time. The composite powders were dried at 150°C for 90 minutes. The resultant powders were then subjected to uniaxial single action compaction at 140 MPa pressure to prepare billets. Final densification process was accomplished by hot forward extrusion process at 600°C with the reduction ratio of approximately 20:1 to produce dense rods of 6 mm in diameter. In all the specimens, the total weight percent of the Al_2O_3 reinforcement were 10 wt.%. To investigate the effect of the

nano-sized reinforcement addition on mechanical properties of the composites, different ratios of the nanometric Al_2O_3 powder to the submicrometric Al_2O_3 powder were tested, including 2:8, 3:7, 4:6, 5:5, and 6:4 in weight percent. In this research, aluminum matrix composites reinforced by two sizes of alumina particles (nanometric and submicron sizes) were prepared by wet attrition milling and hot forward extrusion processes. It was found that by increasing the ratio of the nano- to submicron-sized particulates, the relative density first increases and then decreases. The amount of the micro-hardness and strengths of the composites first increases and then decreases by increasing the nanoparticles content. The increase in the mechanical properties was explained by the Orowan bowing mechanism and thermal mismatch between the matrix and reinforcement particles. For the samples having the nanoparticles more than 4 wt.%, a decrease in the strengths was observed, attributed to the agglomeration of the nanoparticles and the formation of a continuous brittle phase along grain boundaries. In addition, the dimension of dimples formed in the fractured surfaces decreases by increasing the nano-sized particulates.

Hafeez and Senthil (2010) used the base material in the present experimental investigation is Aluminium 6063. Yttrium oxide (Y_2O_3) powder of 99.5% purity with a particulate size range of 25–50 nm, and aluminium oxide (Al_2O_3) powder of 99.995% purity with a particulate size range of 40–50 nm were used as the particulate reinforcements. Volume fraction of 1.3% was used as reinforcement level in the present investigation for the synthesis of all systems of composite mixture. Al 6063, Al 6063/1.3 vol.%Al_2O_3, Al 6063/1.3 vol.%Y_2O_3 and Al 6063/0.65 vol.%Al_2O_3/0.65 vol.%Y_2O_3 nano-composites were synthesized using powder metallurgy technique. The synthesis process involved blending of elemental powders of Al 6063 composition and the reinforcement of nano-sized powders in a two station Insmart Systems, laboratory scale high-energy planetary ball mill at 200 rpm for one and half hours. No ball and process control agent were used during the blending process. The blended powder was regarded as the 0 h mechanically milled powder. Powder blends were then subjected to high-energy ball milling (Insmart Systems, Hyderabad, India.) with the following parameters: charge ratio: 10:1 (wt.); powder mass: 30 g; mass of balls: 288 g; ball diameter: 16mm; ball material: hardened stainless steel; no. of balls: 18; plate speed: 150 rpm: vial speed: 300 rpm; vial material: hardened stainless steel; A total of 75 ml of Toluene (PCA) (Sulphur free) was added in each vial to control the process in order to avoid formation of inter-metallic compounds during milling. The constituent powders were milled continuously for 40 h. The high-energy milling time was the time necessary to complete the mechanical alloying process. The milling process was systematically studied by extracting powder samples for every 10 h to know the grain refinement and micro-structural changes. The ball milling experiments were stopped periodically for every 15 min and then resumed for 15 min, in order to avoid significant temperature rise. Powder samples are designated by Al-6063/1.3Al_2O_3, Al-6063/1.3Y_2O_3 and Al 6063/0.65Al_2O_3/0.65Y_2O_3. It was found that the composite powder reinforced with both alumina and yttria results in much better reduction in crystallite size, which is due to the increase in fracture tendency. High-energy wet ball milling method used for the preparation of nano-composite powders with nano particles reinforcement resulted in refined microstructure and randomly oriented interfacial grain boundaries which is the characteristic of particles at steady-state condition. Better particle size distribution was attained in Al6063/Al_2O_3/Y_2O_3 nano-composite powder, as distinguished from individual reinforcements due to the balance between welding and fracture which confirms the attainment of steady-state condition.

Razavi-Tousi et al. (2011) utilized a high purity aluminum powder (particle size:1mm) was separately mixed with 1, 3 and 7 vf% of MR70 alumina ($D_{50} \approx 500$ nm) and alumina nano-powder. In order to calculate the mean particle size of nano-alumina powder, Brunauer–Emmett–Teller (BET) analysis was used. Supposing monosized spherical particles, a particle size of 39 nm was obtained for nano-alumina powder from Eq. (1): d = 6 aS.BET × ρ where d is the particle size, aS.BET is the surface area obtained by BET analysis and ρ is the density. The mixtures were wet milled in a P5 planetary mill for 22 h using stainless steel cup and balls in Toluene media. The ball to powder ratio was 20:1 and the mill speed was maintained at 300 RPM. The produced powders were dried at 100°C. In order to obtain green samples, the powders were pressed by an isostatic press at 1 GPa in the air atmosphere, the green samples were sintered at 640°C for 30, 60, 120, 240 and 480 min in the Ar atmosphere using a tube furnace. The Archimedes method was used to obtain density of the green and sintered samples; the samples were boiled in methanol for 0.5 h and soaked for 0.5 h. Weight of the samples was measured in dry, wet and soaked condition for obtaining the densities. Effect of a uniform dispersion of alumina particles on compaction and sintering behavior of Al matrix nanocomposite was studied. Pressability of nanocomposite powders decreases as the particle size of reinforcement phase decreases or its volume fraction increases. This effect has been explained by the effect of

reinforcement phase on the strength of composite powders. A similar behaviour was observed in the case of sintering behaviour ascribed to the presence of alumina particles at the sources from which matter transfers. Moreover, the inhibited grain growth of Al matrix profoundly decelerated neck growth or coarsening mechanisms, which resulted in stabilization of initial pores.

Martin et al. (2011) used air atomized Al powder of technical purity (99.8%). Nominal diameters $d_{10} = 0.66\mu m$, $d_{50} = 1.31\mu m$, and $d_{90} = 2.51\mu m$ were determined by Sympatec HELOS laser diffraction method. Al powder featured irregular morphology with abundant fine powder particles detached on the surface. Each powder particle represents a single monocrystal. The powder surface area 4.54 m^2/g was determined by using BET method. BET diameter was calculated according $d_{BET} = 6/(BET *\rho_{Al})$, where ρ_{Al} is the density of Al. Due to irregular morphology of powder rather small $d_{BET} = 0.48\mu m$ was obtained. Calculated Al_2O_3 content on as-atomized Al powder, using an average oxide thickness of 3 nm, was found to be 3.6 vol.%. 1.6 wt.% oxygen content on loose powders was determined by hot gas extraction. Loose powder was cold iso-statically pressed (CIP) at 200 MPa with vacuum assistance prior consolidation. As proved by mercury porosimetry, CIP performs exhibited uniform pores distribution with ~78% of all pores found within 32–60nm size and median pore radius 38 nm. Archimedes's density and mercury porosimetry measurements confirmed relative densities of CIP powder preforms to be 89% of theoretical density. CIP preforms were compacted by direct extrusion (DE), forging (F) or hot isostatic pressing (HIP). DE was performed at an extrusion temperature of 450°C, average ram speed ~1mms−1 using extrusion ratio R = 11:1. Extrusion pressure monitored during DE consolidation reached a breakthrough value of 986 MPa. F was performed using a spindle press at 450°C, 90 kJ of press energy and a maximam speed of 550 mms−1. No powder surface treatment was done prior DE and F. Vacuum degassing of canned CIP preforms at 350°C for 24 h was performed prior HIP. Once sufficiently low vacuum (~10 Pa) was reached, HIP at 500°C under 100MPa pressure for 2 h dwell was performed. Alternatively to HIP, vacuum sintering of CIP preforms within temperature range 450–550°C for 12 h dwell time and 10^{-3} Pa vacuum pressure was carried out as well. In order to study the thermal stability of compacted materials air annealing at temperatures of 450°C for 336 h, 500°C for 24 h and 520°C for 24 h was applied. It was shown, that different powder metallurgy processing routes (extrusion, forging, and HIP/sintering) strongly affect the properties of subsequent fine ($d_{50} = 1.3\mu m$) atomized Al 99.8% powder compacts. It was mainly due to the different distribution, morphology and interconnectivity of Al_2O_3 present on the surface of as-atomized powders after compaction. Extrusion and HIP disrupted native Al_2O_3 layer into separate nano-metric dispersoids found along compact grain boundaries. Forging yielded composite structures of polyhedral Al grains with a continuous interpenetrating Al_2O_3 skeleton. All compacts showed long-term thermal stability and enhanced mechanical properties up to 450°C. On top of it, continuous Al_2O_3 skeleton within Al matrix led to superior mechanical properties and creep performance of forged compacts at elevated temperatures up to 400°C. Furthermore, clean metal-metal grain boundaries assured higher room temperature ductility and conductivity of extruded and HIP compacts.

2.1. Casting

Mazahery et al. (2009) in this study, used A356 aluminum alloy {(wt.%): 7.5 Si, 0.38 Mg, 0.02 Zn, 0.001 Cu, 0.106 Fe and Al (balance)} as the matrix material while the mixture of nano- Al_2O_3 (alumina) particles with average particle size of 50nm and aluminum particles with average size of 16μm was used as the reinforcements. The powders were mixed in the ratio of $Al/Al_2O_3 = 1.67$ and ball milled in isopropyl alcohol for 20min using WC/Co balls. The mixture was then dried in a rotary vacuum evaporator and passed through a 60 mesh screen. The powder mixtures were cold pressed under 200MPa into samples having 60mm×60mm×60mmdimension. The compacted samples were crushed and then passed through 60 mesh screen. For manufacturing the MMCs, 0.75, 1.5, 2.5, 3.5 and 5 vol.% Al_2O_3 particles were used. The required amount of Al_2O_3 was calculated according to the ratio of Al/Al_2O_3. The metal matrix composites have been produced by using a vortex method. Approximately, 450 g of Al-356 alloy was charged into the crucible made from graphite, and heated up to 800°C (above the alloy liquidus temperature) for melting. The graphite stirrer fixed on the mandrel of the drilling machine was introduced into the melt and positioned just below the surface of the melt. Approximately, 1g powder mixture was inserted into an aluminium foil by forming a packet. The packets were added into molten metal of crucible when the vortex was formed at every 20s. The packet of mixture melted and the particles started to distribute around the alloy sample. It

was stirred for 15 min at approximately 600 rpm speed. Composite slurry was poured into preheated cast iron moulds. The composites were shaped in the form of cylinder of 14 mm outer diameter and height of 140 mm. SEM micrographs indicate that nano- Al_2O_3 particles are homogeneously dispersed throughout the composite samples. The grain size measurements show that the grain size of aluminium composite is smaller than that of monolithic aluminium. Porosity level increased slightly with increasing particulate content. These results can be attributed to the increased surface area of the nano- Al_2O_3 particles which can in turn increase the porosity levels. The yield strength, UTS and ductility of nano- Al_2O_3 reinforced aluminium composites improved with the increase in volume fraction of nanoparticles. The great enhancement in values of UTS observed in this experiment is due to small particle size and good distribution of the nano- Al_2O_3 particles, effective transfer of applied tensile load to the nano- Al_2O_3 particulates and grain refinement of aluminium matrix. The hardness of the MMCs increases with the volume fraction of particulates in the alloy matrix due to the increasing ceramic phase of the matrix alloy. The higher hardness of the composites could be attributed to the fact that nano- Al_2O_3 particles act as obstacles to the motion of dislocation. The maximum hardness was observed in composite including 2.5 vol.% Al_2O_3 and cast at 800°C.

Mula et al. (2009) ball milled a micron sized (average size 75 μm) Al_2O_3 powder for 22 h to produce nano-sized (average size ~10 nm) Al_2O_3 dispersoids in a high energy Fritsch Pulverisette- 5 planetary ball mill with WC grinding media. The mill was operated at 300 rpm and toluene was used as the process control agent. The ball to powder weight ratio was maintained at 10:1. In order to minimize/eliminate any possible contamination from the WC grinding media, the WC balls and vials were pre-coated with Al by milling pure Al powder for 4h under identical condition. Since the milled) Al_2O_3 were to be used for the reinforcement of Al-matrix, the Al contamination of) Al_2O_3 was not detrimental to the nanocomposite. The particle size of the milled Al_2O_3 powder was examined by TEM. Commercially pure Al (cp-Al) having a nominal composition of Al–0.96% Fe–0.43% Mg–0.26% Si was reinforced with this nano-sized Al_2O_3 by the non-contact ultrasonic casting method to prepare the cast ingots of Al–2% Al_2O_3 nanocomposite of 205 g weight. Fig.3. schematically illustrates the experimental set-up for this casting method.
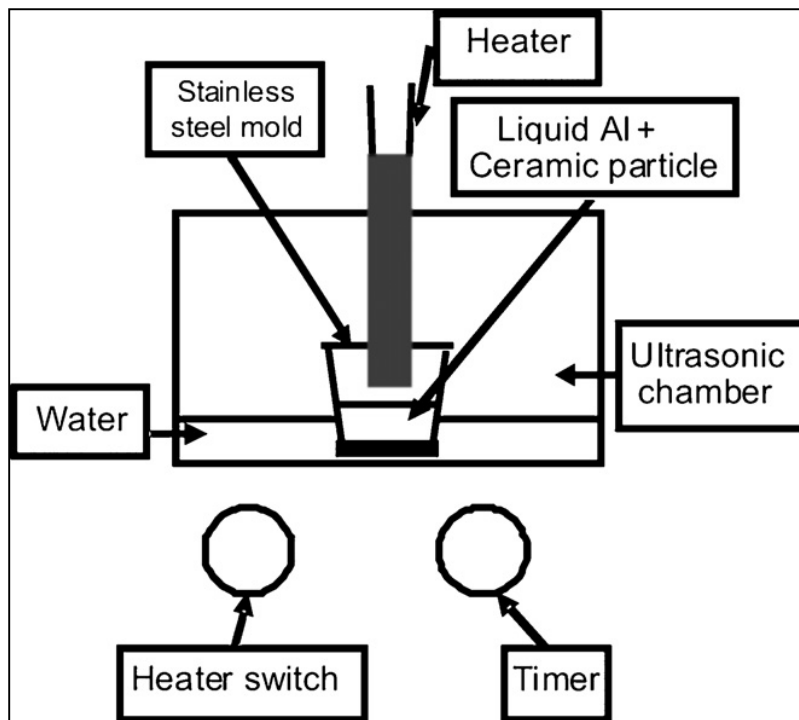

Fig. 3. Experimental set-up for non-contact ultrasonic casting of Al–Al_2O_3 nanocomposite

It consists of an ultrasonic chamber which can vibrate at a frequency of 35 kHz, a stainless steel mold and a hanging heater. A pulley system enabled lowering of this hanging heater into the mold or withdrawing it thereafter. The base of the mold was tightly fitted to the ultrasonic chamber, which was partially filled with water, so that the ultrasonic waves are effectively transmitted to the molten metal. The mold was preheated by the heater to delay the solidification process. After preheating the mold, the heater was withdrawn, and the mold was subjected to vibration at a frequency of 35 kHz. Then the liquid Al and nano-sized Al_2O_3 particles (2 wt.%) were simultaneously poured into the vibrating mold. The heater was immediately brought close to the liquid metal in order to delay the solidification process. The vibration continued for 300 s to ensure completion of the solidification. The ultrasonic waves in the molten metal were expected to generate transient cavitations and acoustic streaming to cause uniform mixing of the nano-sized dispersoids. Investigations on Al–2% Al_2O_3 nanocomposite using optical, scanning and transmission electron microscopes revealed that the nanocomposite was a 'super-composite' of nearly continuous nano-alumina dispersed zones (NDZs) enveloping the Al_2O_3 depleted zones (ADZs). The NDZs were 200–300 nm wide and situated mostly near the grain boundaries. The NDZs had a dense dispersion of nano-sized (~10 nm) Al_2O_3 particles with an average inter particle spacing of ~24 nm. Nearly 92% increase in the hardness and ~57% increase in the tensile yield strength were obtained in the present nanocomposite, as compared to those of the commercially pure (cp) Al, cast by the non-contact ultrasonic casting method. These improvements in the hardness and tensile strength were due to the reinforcement by only 1.4 vol.% nano-sized Al_2O_3 dispersoids in the Al matrix. The standard deviations in the nanoindentation hardness and elastic modulus for the as-cast nanocomposite were higher than those in the ultrasonically cast cp-Al, apparently due to the submicron level inhomogeneous structure of the nanocomposite. Orowan mechanism predicted much higher yield strength than that observed in the present nanocomposite. Some alternative mechanism(s) like local climb and/or cross slip at 0.32TM affecting the strengthening of the present nanocomposite remained a distinct possibility, because the hard Al_2O_3 dispersoids were very fine (~10 nm) even on nanometric scale, and their inter-particle spacing in the NDZs were also small (~24 nm).

Ali and Mohsen (2011) selected aluminum 356 alloy ((wt%): 7.5 Si, 0.38 Mg, 0.02 Zn, 0.001 Cu, 0.106 Fe, and Al as the matrix and a mixture of nano- Al_2O_3 ((wt.%): 93 a-Alumina, 0.8 Fe_2O_3, 1.8 TiO2, 1.1 CaO, and 0.2 other magnetic materials) and aluminum particles with average particle sizes of 50 nm and 16 mm, respectively, were used as the reinforcement. The powders were mixed in the ratio of Al/Al_2O_3=1.67 and they were ball milled in isopropyl alcohol for 20 min using WC/Co balls. The mixture was then dried in a rotary vacuum evaporator and passed through a 60-mesh screen. The powder mixtures were cold-pressed under 200MPa into samples having 60x60x60 mm^3 dimension. The compacted samples were crushed and then passed through 60-mesh screen. The required amount of Al_2O_3 was calculated according to the ratio of Al/Al_2O_3. Magnesium additive (1.0 wt%) in powder form was also used as a wetting agent. The metal matrix composites (MMCs) have been produced using a vortex method. Microstructural observations revealed a reasonably uniform distribution of Al_2O_3 nanoparticles in the Al matrix. These particles refined the grain structure of the cast materials. There are also a few agglomerated particles through the matrix. The addition of nanoparticles resulted in significant improvements in both compressive and tensile flow stress, for which the highest values were obtained at 2.5 and 1.5 vol.% of Al_2O_3 nanoparticles, respectively. The presence of nanoparticles primarily improves the ductility of composite at 800°C. However, the elongation remains rather constant with the addition of nanoparticles at 900°C. Porosity level increased slightly with increasing particulate content at both casting temperatures. These results can be attributed to the increased surface area of the nano-Al_2O_3 particles which can in turn increase the porosity levels. Fractography of the samples displayed lot of dispersed dimples with varying sizes in the matrix, confirming the high ductility observed in the tensile studies.

2.3. Pressure Infiltration

Infiltration process is typically preparing a porous preform‖ of the reinforcement followed by infiltration its pores with the molten metal. Liquid phase infiltration is not straightforward due to the difficulties with wetting the ceramic reinforcement by the molten metal. To overcome the capillary forces that lead to non-wetting, the chemistry of the system must be modified, or an external pressure is applied most to the metal to force the contact and enhance the wettability. Chemical modifications include coating the reinforcement (e.g. with Nickel), adding special elements to

the matrix (e.g. adding Mg), or using special atmospheres and very high temperatures. However, when the infiltration of the preform occurs readily, reactions between the former and the molten metal may take place and significantly degrade the properties of the composite. For this reason, the preform is often coated to reduce the interfacial reactions, thus increasing the complexity and costs of the process.

Timms et. al. (2002) used the equipment for forming 70 mm diameter discs via pressure filtration is shown in figure. The device was positioned upside down relative to that shown in the fig. to allow the air in the system above the slurry to be expelled first. A fine pore size filter paper (2.7 mm) was used to provide the primary resistance to the passage of the powder particles, to build up the initial compact layer of the green body. This was supported by a partially sintered stainless steel disc, having a mean pore size of 6 mm. A typical load versus time trace is given in Fig. 4. showing that the maximum 'dwell' was applied via a low range cyclic load (19.2±0.1 kN; 5 MPa). The crosshead speed used for stages I–IV was 0.5 mm/min, and was 1 mm/min for stage V. Investigations were carried out to deduce the likely origin of such defects and ultimately a humidity cabinet was constructed for controlled drying of the discs. A saturated solution of sodium chloride in water was used to provide a high relative humidity (~75% RH).
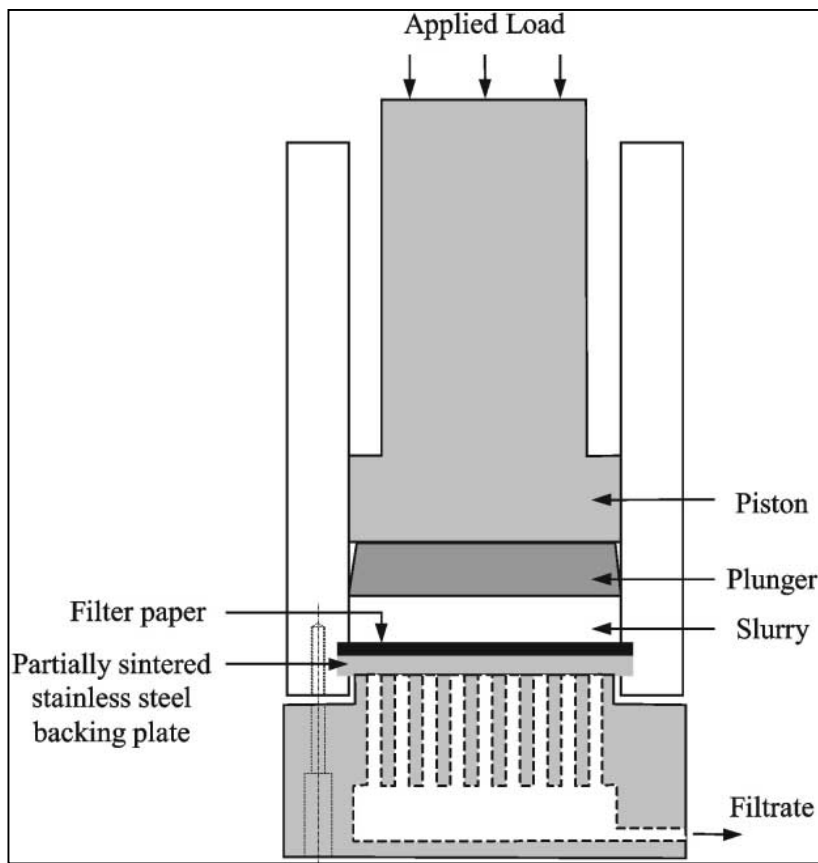

Fig. 4. Schematic diagram of the pressure filtration equipment [Timms et al.(2002)]

Thermo-gravimetric analysis revealed the onset of the dispersant degradation to be around 320°C, with complete decomposition by 540°C. Consequently, a calcinations treatment to 600°C for 2 h was used to remove the polymer prior to sintering. Oxidation of the silicon carbide started at 800°C, giving confidence that no significant oxidation of the second phase particles occurs during the calcination stage. Pressureless sintering of the samples was carried

out in a vacuum furnace, backfilled with argon above 1000°C. Samples were stacked into a graphite pot containing boron nitride powder to keep the samples separated from each other. A heating rate of 5°C/min was used, with a 2 h dwell at the sintering temperature. Cooling was controlled at 10°C/min until the minimum temperature measurable through pyrometer control was reached, followed by furnace cooling (with no power to the elements). Initially different maximum temperatures were used to determine an optimum sintering temperature for the RA207LS/UF15 nanocomposites of 1900°C. Alumina samples were sintered in air at 1600°C with a 2 h dwell, using a heating and cooling rate of 5 C/min. Pressure filtration allows the dispersed sol characteristics to be retained to the point of consolidation, and avoids problems associated with the formation of hard agglomerates when drying slurries; for example, prior to dry pressing. The technique has been used here to produce alumina–5 vol.% silicon carbide nanocomposites with green densities of up to 64% TD. Low green densities are obtained as a result of the practical limits on maximum slurry solids loading when using finer powders. The occurrence of post-forming drying defects can be reduced by controlling the water evaporation rate in a humidity cabinet, with the initial drying rate being dependent on the permeability of the green compact. Pressureless sintering at 1900°C gave near fully dense (99% TD) nanocomposites. Localised sintering took place in the case of the very fine CR15 alumina powder to give very low sintered densities. High sinter-active powders are normally attractive when using pressureless sintering, but problems in processing these powders lead to low green densities in the present work.

Gustafsson et al. (2008) milled a powder mixture of Al_2O_3 (mean particle size 0.4 mm) and 4.1 wt% SiC (; mean particle size 0.45 mm; 2.5 wt% oxygen) in water for 1.5 h with Si3N4 balls. The aqueous suspension had a solid loading of 40 vol%, and contained a dispersant (Dolapix PC 21; 0.35 wt% with respect to the Al_2O_3 and SiC contents). Two doped suspensions were also prepared by adding MgO (0.05 and 0.1 wt% with respect to the Al_2O_3 content) after 1 h of milling. The Al_2O_3: SiC powder weight ratio was chosen so that the sintered material would have a SiC volume fraction of 5%. After milling, the slurries were screened through a 50 mm mesh, and a pressing aid (PEG 400; 3 wt% with respect to the Al_2O_3 and SiC contents) was added. The slurries were then stirred for 1 h, and thereafter screened through a 100 mm mesh. Freeze granulation was subsequently carried out by spraying into liquid nitrogen. The ice was removed by freeze drying and the granules were screened so that the fraction larger than 500 mm was removed. Freeze granulation and drying makes it possible to retain the homogeneity of the aqueous suspension in the ceramic green bodies. The granules were hand-pressed into compacts and these were cold isostatically pressed at 300 MPa. The green bodies were placed in a SiC protective powder bed in a graphite crucible and pressureless sintered in a nitrogen atmosphere for 4 h. Two different sintering temperatures, 1750 and 1780°C, were applied The heating rate of the furnace was 1°C/min up to 600 8C, and then 10°C/min up to the holding temperature. The density of the sintered material was determined by the Archimedean method using distilled water. It is possible to pressureless sinter undoped Al_2O_3–5 vol% SiC nanocomposites to near full density (99.3%) at 1780°C. A smaller addition of MgO (0.05 or 0.1 wt% with respect to the Al_2O_3) is an effective densification aid only at lower sintering temperatures (1750°C), while the density of doped and un-doped materials are virtually the same after sintering at a higher temperature (1780°C). The SiC nanoparticles are well distributed and present at predominantly intra-granular positions. The hardness is strongly dependent on the density, and in the range 17.0–18.5 GPa when the material was sintered at 1780°C. The indentation fracture toughness was 2.3–2.4 MPa m1/2 and did not depend on density, matrix grain size or SiC particle size.

2.4 Wet Chemical Method

Hong-xia et. al. (2005) prepared Al_2O_3 coated Al nano-composite powders by using commercial nanometer Al powders (particle size 80–100 nm, purity 99%), aluminium nitrate (purity of 99%) and ammonia. Aluminium nitrate was first dissolved in distilled water. Appropriate nano-size Al powder was weighted according to a mol ratio Al: Al_2O_3=10:1. The weighed Al powder was added into $Al(NO_3)_3$ solution under vigorous stirring in order to obtain a completely dispersion. Ammonia (diluted to 5.0 vol.% with distilled water) was then dripped into the mixture at a speed of 100 drips per minute. The reaction can be expressed as:

$Al(NO_3)_3 + 3NH4OH \rightarrow Al(OH)_3\downarrow + 3NH_3NO_3$; and $Al(OH)_3$ can be formed on the surface of Al powders. To ensure a complete reaction, excess ammonia was used and the pH value of the solution was maintained at 7–8 during the reaction. Appropriate diluted hydrochloric acid was added in order to prevent agglomeration. The composite deposits were vacuum filtered and washed by distilled water or ethanol for several times. The obtained precipitation

was dried at 70°C for 24 h, and then the obtained precursor was calcined at different temperature in argon atmosphere separately. The composite powders were milled in agate mortar for 2h, pressed into pellets under 15 MPa, and then pressureless sintered at 1450°C for 2h. Al_2O_3-coated Al nano-composites powders were successfully prepared by heterogeneous precipitation using alumiun nitrate, ammonia and nano-size Al powder as starting materials. A uniform thin $Al(OH)_3$ layer can be formed on the surface of Al particle. After calcined at 1000°C for 2 h, the $Al(OH)_3$ layers change to α-Al_2O_3 nano-particles with mean particle size of 10–20 nm and result in a well dispersed alumina-coated Al composite powder with spherical shape. The advantage of the Al_2O_3-caoting Al powder is that a fine transition layer may be formed between Al and Al_2O_3, which can be improved the bonding between Al and Al_2O_3. In addition, due to the close coating of Al_2O_3 on the Al surface that separate Al particles, there is no obvious grain growth of Al particles during the heat treatment. This is desirable for preparing Al_2O_3–Al composite ceramics.

Hongxia et al. (2005) used the nano-size aluminium with purity of 99% in this work. The nano-size aluminium particles have ball appearance approximately with an average grain size about 100 nm. The procedure for preparing plate-like nano-size α- Al_2O_3 powders is shown in Fig. 5.
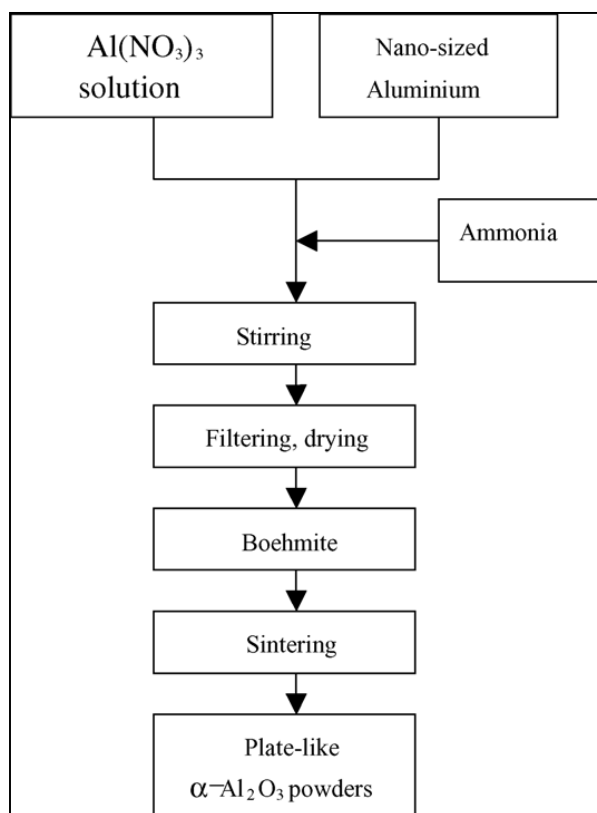

Fig.5. Flow diagram of the procedure used for preparation of plate-like α-Al_2O_3 particles, Hongxia et al. (2005)

Aluminium nitrate was first dissolved in distilled water (0.4 mol l−1). Then 2 mol% nano size aluminium powder was added into $Al(NO_3)_3$ solution under vigorous stirring in order to obtain a complete dispersion. Ammonia (diluted to 5 vol.% with distilled water) was then dripped into the mixture at a speed of 100 drips min^{-1}. Appropriate diluted hydrochloric acid was added in order to prevent aggregation. The pH value of the solution was maintained at 6 during the reaction. The composite deposits were vacuum filtered and washed by distilled water or ethanol for several times. The obtained precipitation was dried at 80°C for 24 h, and then the obtained precursor was calcined in air from room temperature to 1100°C at a constant heating rate of 5°Cmin^{-1}. Nano plate-like α-Al_2O_3 particles with

average size of 80 nm and length of 300 nm can be prepared with nano-size aluminum additives by precipitation method. These nano-size aluminum additives can form Al_2O_3 seeds and liquid phase at the grain boundary enhancing the anisotropic growth and the formation of plate-like α- Al_2O_3 grains. Aluminium additives can also reduce the formation temperature of α- Al_2O_3 to1100°C.

2.5 Friction Stir Process

In Friction Stir Process, a cylindrical rotating tool with a concentric pin and shoulder is plunged into the material surface. Localized heating is produced by friction between the rotating tool and the work piece to raise the local temperature of the material to the hot working range where plastic deformation can be done easily. When the appropriate working temperature is reached, the tool is traversed along the line of interest. Due to stirring action of the pin tool, metal flows to its back side where it is extruded/forged, consolidated, and cooled under hydrostatic pressure conditions

Shafiei-Zarghani et al. (2011) used an A6082 commercial Al–Mg–Si alloy extruded bar as the substrate material. Work pieces were prepared with a thickness, width, and length of 7, 75, and 200mm, respectively. As hallow groove was machined thorough the centre surface of the substrate with a width, length, and depth of 1, 160, and 4 mm, respectively. Nano-sized Al_2O_3 powder with an average particle size of ~50nm was filled in the groove. The simplified FSP unit was a modified form of a conventional miller machine. A hardened H-13 tool steel was used that consisted of a shoulder with a diameter of 16mm and a pin with a diameter and length of 5 and 4mm, respectively. The shoulder tilt angle was fixed at 3°. The tool rotation rate was adjusted at 1250 rpm, and the rotating tool was traversed at a speed of 135 mm/min along the long axis of the work piece. Substrates were subjected to various numbers of FSP passes from one to four. In this investigation, an increase in number of FSP passes causes a more uniform dispersion of fine cluster of Al_2O_3 particles. A good dispersion of nano-sized Al_2O_3 particles was achieved in the surface composite layer produced by three and four FSP passes. An increasing in number of FSP passes result in an increase in the hardness value of SCLs due to more uniform distribution of alumina particles and also decreasing the matrix grain size; a maximum average micro hardness value of 312 HV was achieved for the SCL produced by four FSP passes. Hardness and wear resistance of the SCL produce by four passes was found to be superior to those of the as-received Al substrate. The superior wear behaviour of the fabricated SCL is attributed to increased micro hardness value, presence of hard ceramic particles and matrix grain refinement. The magnitude of improvement in wear resistance of the SCL over the unreinforced Al substrate increases with increase in the applied load. In other words, the difference in wear rate is more pronounced at higher applied loads. At a relatively lower applied load, SCL produces by four FSP passes exhibited mild wear regime. However, it is changed to slightly severe wear at higher applied loads.

3. Comparison of Various Processing Techniques

Process	Advantages	Limitations
Powder Metallurgy	1. Homogeneity of mixture is better controlled, component is produced in near net shape dimension, good ductility, low ball to powder charge ratio provides a better blend homogeneity 2. The gas atomized aluminium particles exhibit a spherical shape with broad size distribution while small satellite particles attached to the large ones 3.The most economical method for manufacturing aluminium MMCs, one can avoid the segregation and agglomeration of the reinforcement particles 4. The high-energy ball milling offers grain size refinement, making the crystals less susceptible to fracture, and hence nano crystallization process of aluminium MMCs has been the subject of intensive research in recent years.	1. In processing of Aluminum, the oxide and hydroxide films coating the powder 2. Metal powders do not act as perfect liquids under pressure and a difference in pressure is established both parallel and perpendicular to the direction of pressing.
Casting	Better matrix–particle bonding, easier control of matrix structure, simplicity, low cost of processing, and nearer net shape	Extremely difficult for the mechanical stirring method to distribute and disperse nano-scale particles uniformly in metal melts due to their large surface-to-volume ratio and their low wettability in metal melts,
Pressure Infiltration	This method allows the powder particles to be kept in a liquid dispersing medium right up to the point of particle–particle contact during the consolidation step, It avoids problems associated with the formation of hard agglomerates when drying slurries	Abnormal grain growth was noted for samples containing the larger particle size, since the number of particles reduces with increasing particle size. This lowers the potential for grain boundary pinning during sintering, and hence, for limiting grain growth.
Wet chemical method	1.The phase of such obtained powder is more uniform, the surface bonding between metal and ceramic was enhanced, and the green density is improved greatly 2. Cost less process for preparing high quality nanosize alumina powders	
Friction Stir Process	1. To form ultrafine-grained structure in Al and Mg alloys 2.To produce a fine-grained microstructure, which exhibits super plasticity 3.To homogenize the microstructure of nano-composite aluminum alloys 4.To refine the microstructure of cast aluminum alloys	

4. Comparison of Various Properties

Process	Process parameters	Properties
Powder Metallurgy	1.Compaction stress of 74-157 MPa and compaction temperature of 723-873K applied for 4 h-3h, followed by hot extrusion 2. Aluminum powder was mixed with different volume fraction (1–7 vol.%) of Al_2O_3 powder and sintering was done at 620°C for 2 hours. 3. The Al powder was blended with 5 vol% Al_2O_3 and 1.5 wt% PCA (Stearic acid powder) in a Turbula T2C mixer 4. Nitrogen gas atomized Al powder with mean particle diameter of 49 μm, α-Al_2O_3 nanoparticles with average size of 35 nm, and stearic acid were used as the starting materials. 5. To prevent powder oxidation, the powders were sealed in stainless steel vial under an argon atmosphere during ball milling. The weight ratio of ball to powder was 4:1. Milling of the mixed powders was conducted for 1–8 h 6. Milling has been carried out at 300 rpm using a toluene medium in order to avoid oxidation or sticking of powders on the wall of the vial. 7. Gas atomized aluminum alloy powders with a mesh size of −100μm was mixed with volume fractions of 5, and 15% commercially available SiC and Al_2O_3 powders. The milling time was varied from 10 min to 6 or 8 h. 8. Commercial aluminum powders with particle size smaller than 63 μm and nano-sized α-alumina powder with %99.5 purity and average size of about 27-43 nm have been provided 9. Commercial purity Al powder as a monolithic system and a mixture of Al–20 wt.% alumina powder were separately milled in a P5 planetary mill for various periods of time up to 25 h 10. A mixture of commercial aluminum (99.7% purity and particle size of 50–70μm) and 15.8 wt% ZnO powders (99.9% purity and particle size of 250 nm) was milled in planetary ball mill in order to produce Al–13.8 wt%Zn/5 vol% Al_2O_3 nanocomposite. 12. The primary materials air atomized commercial pure aluminum powder with a mean particle size of 45 μm and spherical pure alpha- Al_2O_3 powders with average particle sizes of 35 nm (Nanostructured & Amorphous Materials, Inc) and 0.3 μm (AMPCO). 13. The base material used in the present experimental investigation is Aluminium 6063 (Al-6063), whose nominal chemical composition (wt.%), purity and mesh size of the pure elemental powders 14. A high purity aluminum powder (Aldrich, no.: 518573, flakes, particle size: 1mm) was separately mixed with 1, 3 and 7 vf% of MR70 alumina (D_{50} ≈500 nm) and alumina nano-powder. 15. Air atomized Al powder of technical purity (99.8%) was supplied by the company New materials development G.m.bH. Nominal diameters d_{10} = 0.66μm, d_{50} = 1.31μm, and d_{90} = 2.51μmwere determined by Sympatec HELOS laser diffraction method.	1. strength improvement of 64 to 100% compared to the matrix material, considerable ductility, main fracture mechanism in Al-Al_2O_3 MMC is the ductile mode of void initiation, growth, and coalescence, 2. The strengths of composites increased with the 4% volume fraction of particulate. 3. The bulk density of composite powders was found to be higher than that of unreinforced Al, longer milling time was found necessary to achieve steady-state condition compared to microcomposite powder. 4. The compressibility of the lended and milled aluminum/nanometric alumina particles exhibits the same features, the addition of hard nanoparticles to aluminum powder by mixing improves the densification capacity 5. The flexural strength of the sintered specimen (1.2 GPa) using 8 h milled powder was about four times as the sintered specimen (300MPa) using as-mixed powder. 6. An addition of the ceramic reinforcement such as Al2O3 particles improves the wear resistance of the composite 7. Higher hardness is obtained when the size of the reinforcement is small and its volume fraction is high. HEM process decreased the crystallite size of the aluminium matrix to about 45 nm. 8. Strength and hardness were increased by creasing of milling time but the elongation was almost constant, the uniform distribution of ultra-fine alumina particles inhibits stress concentration and crack growth. 9. The effect of the nanostructured Al matrix, the presence of submicron alumina particles and nanometeric particles caused by decomposition of PCA increase the hardness of powder up to 180 HV. 10. The produced nanocomposite had a good thermal stability at temperatures below 400 ∘C. 11.The Al_2O_3/Al nanocomposite powders hardness is near five times higher than pure un-milled Al. A decrease in the Al_2O_3 particle size from 400 to 4 nm has increased the nanocomposite powder hardness of 11%. 12. For the samples having the nanoparticles more than 4 wt.%, a decrease in the strengths was observed, attributed to the agglomeration of the nanoparticles and the formation of a continuous brittle phase along grain boundaries. 13.Addition of ceramic nano particles into the aluminium matrix sustains the crystallite size reduction and better particle size distribution 14. Pressability of nanocomposite powders decreases as the particle size of reinforcement phase decreases or its volume fraction increases. 15. On top of it, continuous Al_2O_3 skeleton within Al matrix led to superior mechanical properties and creep performance of forged compacts at elevated temperatures up to 400 °C.
Casting	1. In this study, A356 aluminium alloy ⨍(wt.%): 7.5 Si, 0.38 Mg, 0.02 Zn, 0.001 Cu, 0.106 Fe and Al	1. Porosity level increased slightly with increasing particulate content, The yield strength,

(balance)⟩ was used as the matrix material (16μm) while nano-Al_2O_3 (alumina) particles with average particle size of 50nm was used as the reinforcements, Al/ Al_2O_3 = 1.67, the crucible temperature upto 800 °C

2. Micron sized (average size ~75 mm) Al_2O_3 powder (Loba Chemie) was ball milled for 22 h to produce nano-sized (average size~10 nm) Al_2O_3 dispersoids in a high energy Fritsch Pulverisette- 5 planetary ball mill with WC grinding media.

3. Aluminum 356 alloy ((wt%): 7.5 Si, 0.38 Mg, 0.02 Zn, 0.001 Cu, 0.106 Fe, and Al (balance)) was selected as the matrix and a mixture of nano-Al_2O_3 ((wt.%): 93 a-Alumina, 0.8 Fe2O3, 1.8 TiO2, 1.1 CaO, and 0.2 other magnetic materials) and aluminum particles with average particle sizes of 50 nm and 16 mm, respectively, was used as the reinforcement

UTS and ductility of nano-Al2O3 reinforced aluminum composites improved with the increase in volume fraction of nanoparticles, The maximum hardness was observed in composite including 2.5 vol.% Al_2O_3 and cast at 800 °C.

2. Nearly 92% increase in the hardness and ~57% increase in the tensile yield strength as compared to those of the commercially pure (cp) Al, cast by the non-contact ultrasonic casting method with 1.4% Al_2O_3 dispersoids

3. The addition of nanoparticles resulted in significant improvements in both compressive and tensile flow stress, at 2.5 and 1.5 vol.% of Al_2O_3 nanoparticles, respectively. Porosity level increased slightly with increasing particulate content at both casting temperatures of 800°C & 900°C.

Pressure Infiltration	1. Pressureless sintering of the samples was carried out in a vacuum furnace (Lenton Thermal Designs Ltd,UK), backfilled with argon above 1000°C. 2. A powder mixture of Al_2O_3 (AKP 30, Sumitomo, Japan; mean particle size 0.4 mm) and 4.1 wt% SiC (UF 25, H.C. Starck, Germany; mean particle size 0.45 mm; 2.5 wt% oxygen) was milled in water for 1.5 h with Si3N4 balls. The aqueous suspension had a solid loading of 40 vol%, and contained a dispersant (Dolapix PC 21; 0.35 wt% with respect to the Al_2O_3 and SiC contents)	1. Pressureless sintering at 1900°C gave near fully dense (99% TD) nanocomposites 2. The hardness is strongly dependent on the density, and in the range 17.0–18.5 GPa when the material was sintered at 1780°C, The indentation fracture toughness was 2.3– 2.4 MPa m1/2 and did not depend on density, matrix grain size or reinforcement particle size.
Wet chemical method	1. Al_2O_3 coated Al nano-composite powders were prepared by using commercial nanometer Al powders (particle size 80–100 nm, purity 99%), aluminium nitrate (purity of 99%) and ammonia and pressureless sintered at 1450°C for 2 h. 2. The nano-size aluminum with purity of 99% used in this work was provided by Jiyuan Nano-Technology Corporation (Henan, China). The nano-size aluminum particles have ball appearance approximately with an average grain size about 100 nm.	1. A uniform thin $Al(OH)_3$ layer surrounding Al particles, which after calcined at 1000°C for 2 h, transforms to α-Al_2O_3 with mean size about 20 nm resulting in well dispersed Al_2O_3–Al composite powder, composite particles have spherical shape with a size about 130 nm and the coating α-Al_2O_3 powder has a size of about 20 nm. 2. Nano-size aluminum additives can form Al_2O_3 seeds and liquid phase at the grain boundary enhancing the anisotropic growth and the formation of plate-like α- Al_2O_3 grains also reducing formation temp. to 1100°C.
Friction Stir Process	An A6082 commercial Al–Mg–Si alloy extruded bar was used as the substrate material. Work pieces were prepared with a thickness, width, and length of 7, 75, and 200 mm, respectively	An increasing in number of FSP passes result in an increase in the hardness & wear resistance value of SCLs due to more uniform distribution of alumina particles and also decreasing the matrix grain size; a maximum average micro hardness value of 312HV was, maximum with 4 FSP

5. Future Scope

Metal matrix composites with micron-size reinforcements have been used with great success in the automotive, aerospace, defense, sports and industrial applications. In case of MMNC's, incorporation of as little as one volume percentage of nano size ceramic particles has lead to a much greater increase in the strength of aluminium. Such potential improvements have great implications for the automotive, aerospace, and, in particular, defense industries due to the drastic weight savings and exceptional properties that can be achieved. Potential aerospace applications may include ventral fins for aircrafts, as well as fan exit guide vanes for commercial airline jet engines which needs

high stiffness and strength, low weight as well as resistance to erosion from rain, airborne particulates and hail. Components used in the automotive industry includes brake system components requiring high wear resistance and thermal conductivity, intake and exhaust valves requiring high creep resistance and resistance to sliding wear, as well as piston liners requiring high wear resistance, good thermal conductivity and low coefficient of thermal expansion. Metal matrix nanocomposites can be designed to reveal high thermal conductivity, low density, and matching coefficient of thermal expansion with ceramic substrates and semiconductors, making them ideal candidate for such applications.

6. Challenges

In the area of nanocomposites

1. Problems with non-compatibility and agglomeration, which can be overcome through surface modification of reinforcements for homogeneous dispersion without agglomeration
2. Research on structure-property correlations in nanocomposites, new challenges in the development of suitable fabrication techniques for nanocomposites, their characterization and mechanics, in order to understand interactions at such small sizes.
3. To control the grain size of matrix and agglomeration of nano particles during the processing and retaining the improved microstructure.
4. The release of nanoparticles into the environment is a major health and safety issue which needs further research.
5. There is need to improve the damage tolerance properties particularly fracture toughness and ductility.
6. Research is expected to produce high class and low cost reinforcements from industrial wastes and by-products.
7. There is a greater need to categorize different grades of AMNCs based on property profile and manufacturing cost

7. Conclusions

There are exhilarating opportunities for producing extraordinarily strong, light weight, wear resistant metal matrix nanocomposites with adequate ductility by solidification processing and powder metallurgy. A fundamental understanding, however, must be gained of the mechanisms that provide these improvements in properties, if such materials are to find wider commercial applications. Moreover, processing methods must be developed to synthesize these materials in bulk, at lower cost, with little or no voids or defects, and with improved ductility, possibly as a result of bimodal and tri-modal microstructures. Metal matrix nanocomposites can lead to significant savings in materials and energy and reduce pollution through the use of ultra-strong materials that exhibit low friction coefficients, high wear resistance, low coefficient of thermal expansion and light weight.

1. A detail study of the work done so far has been done in this paper on various processing techniques and characterization of Al nano Al_2O_3 composites.
2. A comparison of properties has also been done for the materials fabricated by different techniques to select a nanocomposite material required for a particular application.
3. From the various processes discussed in this paper, the ultrasonic-assisted casting is a method which provides better matrix–particle bonding, easier control of matrix structure, simplicity and low cost of processing.
4. Mechanical stirring method, it is exceptionally difficult to distribute and disperse nano-scale particles uniformly in metal melts due to their large surface-to-volume ratio and their low wettability in metal melts.
5. It is reported that mechanical properties of Aluminum alloy can be enhanced by reinforcement of nano Alumina particles up to the 4.0 vol.% and beyond that strengthening effect is leveled off because of clustering

of nano Al$_2$O$_3$ particles which requires further research in this area to produce components with high strength and hardness.

6. Further research is necessary to develop better processing techniques which could control and retain properties and microstructures of nanocomposites, better considerate of microstructure and properties and to develop new potential materials for the aerospace, automobile, defense and other industrial applications.

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