SYNTHESIS AND CHARACTERIZATION OF ALUMINIUM-BASED NANOCOMPOSITE REINFORCED WITH BALL MILLED COAL FLY ASH

MAJOR PROJECT – II

SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS
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MASTER OF TECHNOLOGY

IN

PRODUCTION ENGINEERING

Submitted by:

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CANDIDATE'S DECLARATION

I, Hitesh, Roll no. 2K18/PIE/04 student of M. Tech. (Production Engineering), hereby declare that the project dissertation titled **"Synthesis and Characterization of Aluminium-based Nanocomposite Reinforced with Ball Milled Coal Fly Ash"** which is submitted by me to the Department of Mechanical Engineering, Delhi Technological University, Delhi in partial fulfillment of the requirement for the award of the degree of Master of Technology in Production Engineering, is original and not copied from any source without proper citation. This work has not previously formed the basis for the award of any Degree, Diploma Associateship, Fellowship or other similar title or recognition.

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Date: 22.11.2020 | (2K18/PIE/04)

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CERTIFICATE

I hereby certify that the Project Dissertation titled **"Synthesis and Characterization of Aluminium-based Nanocomposite Reinforced with Ball Milled Coal Fly Ash"** which is submitted by **Hitesh (Roll no. 2K18/PIE/04)**, Department of Mechanical Engineering, Delhi Technological University, Delhi in partial fulfillment of the requirement for the award of the degree of Master of Technology in Production Engineering, is a record of bonafide project work carried out by him under my supervision and guidance. To the best of my knowledge this work has not been submitted in part or full for any Degree or Diploma to this University or elsewhere.

Place: New Delhi

Date: 22.11.2020

PROF. (DR.) REETA WATTAL

(SUPERVISOR)

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ABSTRACT

A paradigm shift from monolithic materials to composite materials has been observed in the recent times to achieve the ever changing demands of the manufacturing sector. The composites are being explored in terms of industrial and thermal power plant wastes used as reinforcements. Being a prominent thermal power plant waste, fly ash disposal and utilization is gravitating environmentalists and researchers to propose innovative ideas and feasible implementations. To trace and adjoin an effort to add value to aforementioned cause, an investigation was conducted on fly ash which was milled in tumbler ball mill with predefined input parameters viz. spindle speed as 120 rpm, ball-to-powder ratio as 10:1 (by weight), and milling time was set as 1200 minutes with regular stoppage intervals. Low-energy mill has an extensive application in preparation of new materials, their activation and their process of synthesis for potential merits like cost-effectiveness, reliability, low maintenance and ease of operation over its contemporary high-energy mill. The modifications in chemical and morphological properties of milled fly ash sample were characterized through X-ray diffraction (XRD) technique, scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX). The results inferred that the ball-milling process aimed at reducing crystallite domain size of milled fly ash, and also affected the microstructure, functional groups and led to decline in degree of crystallinity (DOC). The milling time was observed as a significant factor to impact extent of these changes. The particles of the raw spherical-shaped fly ash (regularly 5 μm size) were significantly broken down into smaller particles of average crystallite domain size ~30 nm, this change indicated an increase in amorphousness of fly ash that would help in achieving better compatibility and significantly higher reactivity. The fabrication of aluminium-based nanocomposite (AMC) reinforced with ball milled coal fly ash was done successfully via stir casting route. It was inferred that ductility of said nanocomposite was lowered on adding the ball milled coal fly ash. In order to maintain the ductility, TiO_2 was also added as a reinforcement. The research and analyses defined the achievement of nanostructured fly ash with the low energy mill technique as well as its effective utilization in Aluminium-based composite fabrication and its mechanical characterization.

Keywords: Fly ash, ball milling, stir casting, AMC

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CHAPTER 1
INTRODUCTION

1.1 Nanotechnology: An Introduction

Over the past century, with advancement in technology, there is a subsequent demand of advanced engineered materials from it which has led to increased interest in the field of composites. Researches made in this field has confirmed that a reinforced composite possess not only superior and enhanced thermal and mechanical properties, but stability too. This has resulted in shift from monolithic and pure materials to reinforced matrices [1]. Similarly, shift is visible in the scale and depth at which modifications are being done in the composites too i.e. initially started at macro which progressed to micro with same work being performed mainly on nano-level. Any activity in a structure initiated at nano-scale that is under 100 nm is a part of 'Nanotechnology' [2, 3].

Nanotechnology is the study of the controlling the matter on an atom and molecular scale. Generally nanotechnology deals with structures sized between 1-100 nanometers in at least one dimension, and involve modifying or developing materials within that size. It makes the material lighter, stronger, faster, smaller and more durable. Nanotechnology obligates the ability to frame components of molecular size and precise machine. In other words, 'nanotechnology' refers to the contrived ability to construct items from the bottom up, using tools and techniques that are being defined to make high performance products. In 1959, a physicist R. Feynman envisioned this theoretical capability. According to National science Foundation, Nanotechnology is the capability to understand, manipulate and control matter at the level of individual atoms and molecules [1]. Science and engineering are the primary operators of global technological competition. Modern science based on the unifying features of nature at the nano scale contributes a new foundation for innovation, knowledge, and integration of technology [1]. Nanotechnology is sometimes proffered as a general purpose technology because in its advanced version it will have significant impact on almost all areas of society and all industries [2]. There is a longitudinal process of convergence and divergence in extensive areas of engineering and science. For example the convergence of sciences at macro scale was intended during the Renaissance, and it was latterly followed by narrow disciplinary specialization (NDS) in science and engineering in the 18th-19th centuries. The convergence at the nanoscale reached its brawn in about year 2000, and an estimation of a divergence in the nano system architectures in the next decades. The Figure 1.1 represents how technologies converged to nano particles and how the nano world reached [3].

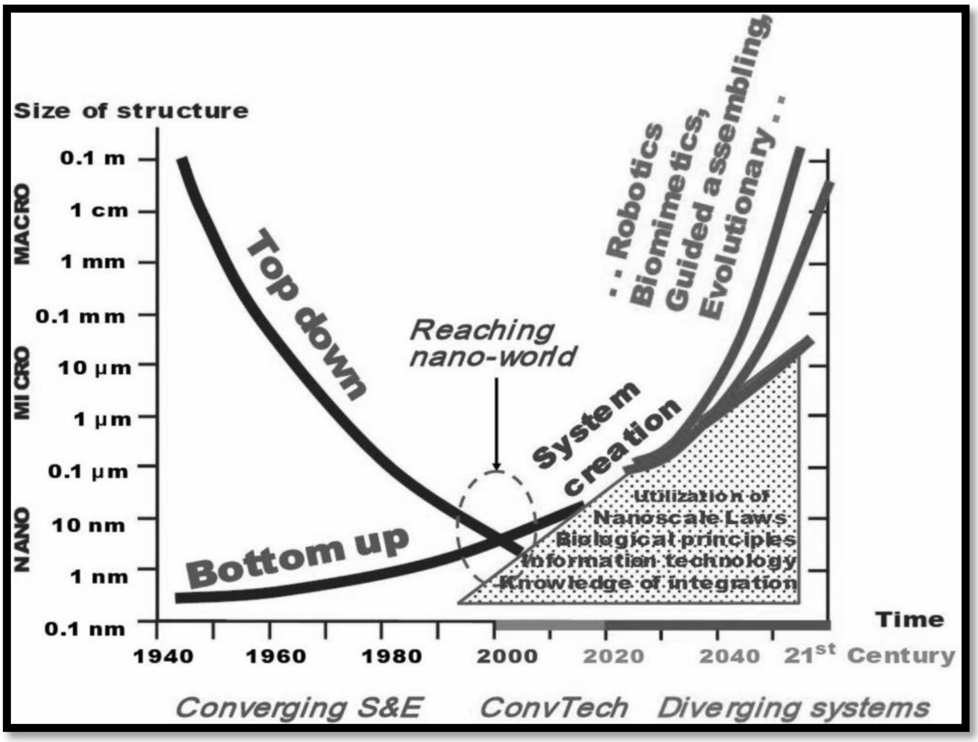

Figure 1.1 Technological approach to nano world [1]

1.2 Need of Nanoparticles

Nanoparticle research is currently an area of intense scientific research, due to a wide variety of potential applications in biomedical, optical, and electronic fields. Nanoparticles are of great scientific interest as they are effectively a bridge between bulk materials and atomic or molecular structures. A bulk material should have constant physical properties regardless of its size, but at the nano-scale this is often not the case. Size-dependent properties are observed such as quantum confinement in semiconductor particles, surface plasmon resonance in some metal particles and super-paramagnetism in magnetic materials. The properties of materials change as their size approaches the nanoscale and as the percentage of atoms at the surface of a material becomes significant. For bulk materials larger than one micrometer the percentage of atoms at the surface is minuscule relative to the total number of atoms of the material. The interesting and sometimes unexpected properties of nanoparticles are not partly due to the aspects of the surface of the material dominating the properties in lieu of the bulk properties. Nanoparticles exhibit a number of special properties relative to bulk material. Nanoparticles often have unexpected visible properties because they are small enough to confine their electrons and produce quantum effects. For example gold nanoparticles appear deep red to black in solution. Nanoparticles have a very high surface area to volume ratio. This provides a tremendous driving force for diffusion, especially at elevated temperatures. The large surface area to volume ratio also reduces the incipient melting temperature of nanoparticles [4].

1.3 Applications of Nanoparticles

1. *Cosmetics and Sunscreens:* The conventional ultraviolet (UV) protection sunscreen lacks long-term stability during usage. The sunscreen including nanoparticles such as titanium dioxide provides numerous advantages. Some lipsticks use iron oxide nanoparticles as a pigment [3].
2. *Electronics:* The higher necessity for large size and high brightness displays in recent days that are used in the computer monitors and television is encouraging the use of nanoparticles in the display technology. For example nanocrystalline lead telluride, cadmium sulphide, zinc selenide and sulphide, are used in the light emitting diodes (LED) of modern displays. The development in portable consumer electronics such as mobile phones and laptop computers led to the enormous demand for compact, lightweight and high capacity batteries. Nanoparticles are the ideal choice for separator plates in batteries. A considerable energy can be stored compared to traditional batteries due to their foam like (aerogel) structure. Batteries made from nanocrystalline nickel and metal hydrides, due to their large surface area require less recharging and last longer [2].
3. *Medicine:* Nanotechnology has improved the medical field by use of nanoparticles in drug delivery. The drug can be delivered to specific cells using nanoparticles [1]. The total drug consumption and side effects are significantly lowered by placing the drug in the required area in required dosage.
4. *Food:* The improvement in production, processing, protection and packaging of food is achieved by incorporating nanotechnology. For example a nanocomposite coating in a food packaging process can directly introduce the anti-microbial substances on the coated film surface [1].
5. *Construction:* Nanotechnology has improved the construction processes by making them quicker, inexpensive and safer. For example when nanosilica (SiO_2) is mixed with the normal concrete, the nanoparticles can improve its mechanical properties, and also improvements in durability. The addition of haematite (Fe_2O_3) nanoparticles increases

the strength of the concrete. Steel is the most widely available and used material in the construction industry. The properties of steel can be improved by using nanotechnology in steel for example in bridge construction the use of nano size steel offers stronger steel cables [1].

1.4 Industrial waste: Fly ash

Fly ash is a waste product generated from coal fired thermal power stations. Large number of coal fired thermal power plants all over the world dispose a huge quantity of the fly ash causing serious environmental problems. During the process of (coal) burning, according to an estimate, about 112 million tons of the fly ash has been presently generated per annum in India alone, whose level is to be further raised with the installation of more new thermal power plants by 2020. Depending upon the source and composition of the coal being burned, the components of fly ash vary considerably, but all fly ash includes substantial amounts of silicon dioxide (SiO_2) (both amorphous and crystalline), aluminium oxide (Al_2O_3) and calcium oxide (CaO).

Figure 1.2 A class-F raw fly ash sample [3]

The fly ash causes great health hazards and environmental pollution, and the situation has worsened to the extent that even dumping of ash is becoming a problem because of the shortage of space required to accommodate the large volume of its generation. As the raw materials are being consumed at a faster rate than they are being replaced, it becomes necessary to think about efficient use of natural resources and reuse and recycling of industrial wastes. The accumulation and disposal of industrial wastes like FA are of national and international concerns [4, 5].

1.5 Aluminium Matrix Composite

Aluminum composites are the forerunners of material development during the last century. They are continually being explored by material design engineers and metallurgist to replace various conventional materials for optimal performance and economic feasibility. Besides having high specific strength, aluminum composites show high specific stiffness, electrical and thermal conductivity, low coefficient of thermal expansion, and wear resistance.

Particulate composites are widely used in composites development because they are cheap and of manufacturing ease. Particulates of fly ash, TiO_2, SiC, Al_2O_3, and Si_3N_4 & B_4C are common reinforcement used in preparing composites [6].

CHAPTER 2
REVIEW OF LITERATURE

2.1 Review of Literature

G. Venkatachalam et al. [7] presented characterization of A356 composite that is produced by stir casting method. Said composite is reinforced with fly ash and basalt ash. Herein, three sets of the composite were prepared by varying weight fraction of aforementioned reinforcements (namely, 3% basalt + 7% fly ash, 5% basalt + 5% fly, and 7% basalt + 3% fly ash). The effect of the reinforcements on mechanical properties of the composite such as hardness (Vickers), tensile, compressive and impact strength were studied. The results revealed that the tensile, the compressive and the impact strength were increased with increase in the weight fraction of the fly ash, whereas the hardness was increased with increase in the weight fraction of the basalt ash.

V. K. Sharma et al. [8] fabricated metal matrix composite (MMC) with aluminum as base metal and fly ash as reinforcement by means of stir casting. Herein, the MMC was fabricated with 2-4-6% weight of the fly ash in aluminum matrix. The friction force and wear of the MMC were investigated by using a Pin-on-disc setup. The wear resistance of the MMC increases with increase in the fly ash content. Specifically, the MMC with high fly ash content resulted in 13.6% less wear as compared to low fly ash content. The results further showed that the MMC with 6% weight of the fly ash content in the aluminum matrix suffered less wear and 4% weight of the fly ash content showed a low coefficient of friction (0.12) between tribo-pairs of cast iron surface and the MMC surface.

B. R. Reddy and C. Srinivas [9] presented a study pertaining to fabrication and characterization of 6082 aluminum matrix hybrid composites with silicon carbide (SiC) and fly ash as reinforcements. Herein, for said fabrication, weight fractions of 2.5, 5 and 7.5 % SiC and fly ash are used in equal proportions. The microstructural properties of the hybrid composites were studied with help of scanning electron microscopy. Moreover, mechanical properties (namely, ultimate tensile strength and hardness) of the hybrid composites were also studied along with dry sliding wear behavior of the hybrid composites using pin-on-disc apparatus. The results revealed an appreciable improvement in the ultimate tensile strength, the hardness and the dry sliding wear behavior as the weight fraction of both the reinforcements was increased gradually.

P. B. Pawar et al. [10] presented a comprehensive study of aluminum based metal matrix composites. Herein, the comprehensive study majorly relates to methods (namely, stir casting, powder metallurgy, squeeze casting, spray deposition, and the like) of preparation of composites, mechanical (such as, strength to weight ratio, tensile strength, toughness, high wear resistance, and the like) and tribological properties of the composites over conventional metals, and potential applications of the composites (such as, a light-weight application of the composites in fields such as, automobile, aerospace, sports, and the like). Moreover, the comprehensive study also relates technological barriers in development of such composites. For example, obtaining uniform distribution of reinforcement among matrix, high processing cost with the methods other than casting, availability of low cost reinforcement and machining of the composites after preparation. The comprehensive study further relates to hybrid composite with fly ash, lignite ash, coconut husk that have provided better results than a single reinforcement.

F. Nturanabo et al. [11] presented a comprehensive study pertaining to development, utilization and future potential of aluminum metal matrix composites in different industrial applications. The comprehensive study revealed that advanced technological developments in processing of the aluminum metal matrix composites will continue to give a competitive edge over alternative materials such as Mg, AHSS and polymer composites with challenges and barriers (such as, lack of property modelling, especially high-temperature behavior of the aluminum metal matrix composites, lack of design data and high costs of processes). The comprehensive study further revealed that with emergence of new developments in CNT and nanotechnology, fabrication of the aluminum metal matrix composites with enhanced properties for high-temperature application, improved wear resistance and improved corrosion resistance have promising possibilities. Developments such as, rheocasting process of semi-solid alloys and FGMs may provide cost reduction in processing of the aluminum metal matrix composites. The comprehensive study still revealed that new alloys of aluminum have been developed areas like crash management as crash alloy that was formerly dominated by steel. Moreover, it was appreciated that various researchers are providing suggestive efforts to bring down cost of replacing conventional ferrous materials with the aluminum metal matrix composites.

R. Manimaran et al. [12] reviewed on mechanical properties of fly ash composites. The review revealed that characteristics of a lightweight composite material such as, fly ash particles, with higher hardness, higher strength, and improved stiffness is gaining popularity. The fly ash particles have proved its potential as good reinforcement with aluminum alloys and show the improvement of physical and mechanical properties. Particularly, compression strength, tensile strength, and hardness increase when the percentage of fly ash content is likely to be increased, whereas the density decreases. The reviewed identified gaps in the study of the fly ash particles for impact strength, corrosion resistance, wear resistance, microstructure, toughness, and ductility.

S. P. Kumarasamy et al. [13] investigated analysis and characterization of hybrid aluminum 7075 metal matrix composite that was fabricated by means of two-step compo-casting technique. Herein, said composite utilized fly ash cenosphere (10%) and graphite (2%, 4% and 6%) as a reinforcements. Results showed that hardness and tensile strength were increased with addition of cenosphere and decreased with addition of graphite. Particularly, the reinforcements enhanced the tensile strength of the aluminum from 178 MPa to 213 MPa. Moreover, the addition of cenosphere and graphite considerably improves wear resistance and wear rate. Furthermore, turning characteristics of the metal matrix composite were studied and optimized by employing Artificial Neural Network (ANN) technique. Said study revealed that cutting speed parameter and percentage addition of graphite have majorly contributed to minimizing surface roughness of said composite.

A. Kamble and S. G. Kulkarni [14] investigated microstructural examination of waste sugarcane bagasse ash (SCBA) reinforced in scrap aluminum matrix. Such a composite was fabricated by stir casting method. Herein, weight percent of reinforcement (namely, SCBA) was varied from 0% to 2.5%. The examination showed that microstructure of fabricated composite had a uniform distribution of the SCBA particles in the scrap aluminum matrix. It was also observed that hardness properties were increased with increase in the weight percent of reinforcement.

H. Gireesh et al. [15] fabricated aluminum metal matrix composites with aloe vera powder as a reinforcement material, wherein the aloe vera powder being a less denser and eco-friendly material at a low cost. It was also suggested that aloe vera powder could replace fly ash for providing better physical and mechanical properties. Herein, specimens of the aluminum matrix composite are fabricated by means of stir casting with the fly ash as reinforcement as well as the aloe vera as reinforcement. Further, mechanical characterization of specimens of the aluminum matrix composite was done to reveal a significant improvement in mechanical properties (such as hardness, tensile strength and impact strength) when the aloe vera was used as reinforcement to that of the fly ash.

A. Bhandakkar et al. [16] studied deformation behavior of aluminum alloy AA6061-10% fly ash composites for aerospace application. Herein, constitutive flow behavior of the aluminum alloy AA6061-10% fly ash composites was investigated under hot-working conditions to generate a processing map for optimizing hot workability. The hot deformation behavior of the aluminum alloy AA6061-10% fly ash composites and aluminum alloy AA6061 was investigated by means of hot compression tests and both the hot deformation behaviors were then compared. Moreover, zones of flow instability, microstructural and failure mechanisms pertaining to the hot deformation of the aluminum alloy AA6061-10% fly ash composites were studied under optical microscope and scanning electron microscope.

B. R. S. Kumar et al. [17] investigated mechanical and wear (or tribological) properties of LM24/silicate/fly ash hybrid composite. Herein, the LM24/silicate/fly ash hybrid composite was prepared with 4 weight% fly ash particles and with 4, 8, 12, 16, 20, and 24 weight% of silicate using vortex technique. Said investigation showed effectiveness of incorporating silicate particles in the composite for gaining wear reduction. Moreover, the silicate particles with the fly ash particles were incorporated into aluminum alloy (namely, LM24) matrix to accomplish reduction in wear resistance and improve the mechanical properties. The wear properties were evaluated under diverse loads (such as, between 15-75 N) and sliding velocity (0.75-3 meters per second) condition using pin-on-disc apparatus and mechanical properties (for example, density, hardness, impact strength, and tensile strength) of said composites were also investigated. Additionally, machining of the LM24/silicate/fly ash hybrid composite was studied using Taguchi L9 orthogonal array with analysis of variance.

G Raghavendra et al. [18] studied flexural and tensile properties of micrometer (5, 10 and 15 weight%), and nanometer (2, 4 and 6 wt%) fly ash particle-filled jute/glass hybrid epoxy composites. Later, corresponding properties were compared with conventional fibers (glass) composites. Results revealed that due to microfiller and nanofiller addition, strength of all composites was increased. Tensile strength of 4 weight% fly ash nano-filler hybrid composites were better than that of other composites, whereas 4 wt% fly ash nano-filler hybrid composites had better flexural strength than that of other composites.

N. A. Patil [19] studied effect of SiC/fly ash reinforcement on surface properties of aluminum 7075 hybrid composites. Herein, microstructure, wear resistance under dry sliding conditions and microhardness of the aluminum 7075 was examined by varying tool rotational speed, tool traverse speed, reinforcement's hybrid ratio, and volume percentage. By using response surface methodology (RSM), desirable ranges of process parameters for lower wear rate and higher microhardness were obtained. Also, for stirring parameters, tool traverse speed was found to be more influential than tool rotational speed. Furthermore, reinforcement played a major role in controlling surface properties of resultant composites.

S. P. Kumar et al. [20] studied microstructure, mechanical and wear properties for stir casting produced Al7005 matrix alloy and S-glass + fly-ash hybrid MMCs with different weight fractions. The microstructure of the hybrid MMCs showed a relatively uniform distribution of S-glass and fly-ash in Al matrix. It was also revealed that mechanical and tribological properties of the hybrid MMCs were improved with increasing weight % of S-glass and fly-ash.

M. Ramachandra [21] investigated effect of reinforcement of fly ash on sliding wear, slurry erosive wear and corrosive behavior of aluminum matrix composite. Herein, aluminum was employed (with 12 wt% Si) as matrix material up to 15 wt% of fly ash particulate for fabricating composite using stir casting route. It was noted that fly ash improved abrasive wear resistance (20-30%) of Al and reduced coefficient of friction. Further, an increase in normal load and sliding velocity increased magnitude of wear and frictional force. Different wear mechanisms were also studied by varying different parameter such as normal load, percentage of fly ash content and sliding velocity. It was also concluded that corrosion resistance of reinforced composite was decreased with increase in fly ash content.

Sudarshan and M. K. Surappa [22, 23] synthesized A356 Al–fly ash particle composites and studied mechanical properties and dry sliding wear of the composites. It was observed that fly ash with narrow size range (53-106 μm) show better properties when compared to wider size range (0.5-400 μm) particles. Further, it was observed that damping capacity of the composite increased with increase in volume of fly ash and fracture surface of composites showed mixed mode (ductile and brittle) fracture. Moreover, 6% of fly ash particles into A356 Al alloy showed low wear rates at low loads, while 12% of fly ash particles into A356 Al alloy showed lower wear rates as compared to unreinforced alloy in the load range 20N to 80N. At higher load, subsurface delamination was the main mechanism in both alloy as well in the aforesaid composites.

R. Q. Guoa and P.K. Rohatgi [24] investigated changes of chemical reaction between the Al and the fly ash during synthesis or reheating of fly ash. Herein, it was observed that the chemical reactions occur between the fly ash an Al melts. The silicates and ferrites present in the fly ash is reduced to Si and Fe. It was also observed that said reactions occur rapidly at the temperature more than 8500 degree Celsius. At this higher temperature, free energy of transformation has significant negative value for said reaction to take place spontaneously.

P. K. Rohatgia et al. [25] investigated compressive characteristics of A356/fly ash cenosphere composites synthesized by pressure infiltration technique and concluded that using gas pressure infiltration, up to 20 to 65% volume percentage of fly ash can be reinforced to A356. Moreover, factors affecting densities of the composites were found to be melt temperature, applied pressure, and size of particles.

P. K. Rohatgi et al. [26] studied coefficient of thermal expansion of pure Al containing 65 vol% of hollow fly ash particles. It was suggested that Al-fly ash composites with a lower coefficient of thermal expansion can be made by incorporating cenosphere(s) while controlling processing parameter for a given volume fraction of reinforcement. It also said that when composite synthesized at different pressure for different infiltration time, increase in the infiltration pressure and temperature improves the infiltration and decreases entrapped air voids which further lower the coefficient of thermal expansion.

J. Sobczak et al. [27] synthesized fly ash particles as reinforcement in metal matrix composites by squeeze casting technology and gravity casting. Herein, a comparison between the aforesaid metal matrix composites was done and it was concluded that the squeeze casting technology is relatively more useful in comparison with the gravity casting. The reason for this was given as the squeeze casting technology has structural homogeneity with less porosity and good interracial bonding. Moreover, in the squeeze casting technology, the composite has a corrosion that was pitting type corrosion.

S. Sarkar et al. [28] studied on aluminum-fly ash composite produced by impeller mixing. Herein, it was observed that up to 17wt% fly ash reinforcement can be used as a reinforcement for liquid metallurgy route. It was appreciated that the addition of magnesium into the aluminum melt increase wettability and increase in mechanical properties such as hardness, tensile strength and the wear resistance.

P. K. Rohatgi et al. [29] studied that Al-fly ash composite can be used for automotive and other applications. The review divulged details on environmental and energy benefits when said composite being used. It also shed some light on the potential cost, energy, and pollution savings as a result of incorporation of fly ash in aluminum. It was appreciated that the potential reduction in cost and energy content of individual auto parts, energy consumption, and emissions due to the replacement of aluminum by Al-fly ash composites.

Narayana Swami et al. [30] fabricated nano structured fly ash by high-energy ball milling. Herein, ball milling was carried for a duration of 30 hours and sample was collected after every five hours for characterizing the nanostructured fly ash obtained from fresh micron fly ash for its crystalline size, lattice strain and percentage crystalline by X-ray diffraction. It was found that after 30 hours ball milling, the crystallite size was reduced from 92 nm to 29 nm and percentage crystallinity was reduced from 63 to 38 percent.

Thomas Paul et al. [31] selected F class fly ash and converted it into nanostructured material through high-energy ball milling process. Herein, particle size, specific surface area and structure of nano structured fly ash was characterized through processes such as, particle size analyzer, BET and (X-ray diffraction) XRD. It was observed that after high-energy ball milling, particle size got reduced from 60 μm to 148 nm by 405 times and surface area increased from 0.249 m^2 /gm to 25.53 m^2 /gm.

Ying Chen et al. [32] synthesized one-dimensional (1D) nanomaterials using high-energy ball milling, wherein two different types of processes were chosen such as ball milling and annealing and a high-energy ball milling (HEBM) that is different from conventional ball milling technique.

Babu Rao et al. [33] modified micro sized silicon carbide powder to nanostructured silicon carbide powder using high-energy ball mill technique. Herein, total duration of ball mill was 50 hours and a sample was taken after every 5 hours of ball milling for characterization in terms of its crystallite size, lattice strain and percentage of crystallinity. It was revealed that after 50 hours, milling the crystallite size was reduced from 120 nm to 26 nm and the percentage of crystallinity got reduced from 74 percent to 49 percent.

Murthi et al. [34] conducted experiments to modify micro-sized fly ash into nanostructured fly ash using high-energy ball mill process. Furthermore, samples of the nanostructured fly ash were taken for characterization and it was revealed that the nanostructured fly ash with AA2024 matrix improved so formed composite's hardness and compressive strength.

Simoes et al. [35] investigated influence of dispersion technique for producing carbon nano tubes (CNT) in production of aluminum matrix nanocomposites. It was observed that addition of 0.75 % by wt. of CNT to aluminum matrix, manifested a well dispersed composition of CNT in the matrix, and said manifestation imparted a high hardness and a high tensile strength to the aluminum matrix nanocomposites.

M. N. E. Efzan [36] investigated microstructure of prepared aluminum matrix composites (AMCs) with the consistent distribution of fly ash was analyzed using an optical microscope. Herein, the microstructure of the prepared aluminum matrix composites microstructure had a refinement of structure with decreasing of Si-needle structure and increasing area of eutectic α-Al matrix. Furthermore, it was also revealed that by increasing amount of fly ash, there were more petal-like dark structure in the microstructure, along with this, a decline in density of the AMCs was also observed. It was concluded that hardness and ultimate tensile strength of the AMCs was increased with increasing wt% of fly ash. It was further concluded that with addition of the fly ash particles, physical and mechanical properties of the AMCs were improved and thereby improving an energy consumption in automotive parts.

2.2 Identified Gaps in Literature

The literature review was conducted in spirit to identify following gaps:

1. The currently available literature explored a limited area of application for utilization of coal fly ash, such as being limited to cement-based products (for example, poured concrete, concrete block, brick, and the like).
2. The exploration of low-energy mill (tumbler mill) over its contemporary high-energy mill (vibratory and planetary mills) is scarce in the currently available literature. However, this kind of low energy mill may lead to an increase in the required milling time for a complete mechanical alloying process. The low-energy mill produces homogeneous and uniform powders. In addition, it is cheaper than those of the high-energy mills and can be self-made with lower costs. Moreover, tumbling mills are operated simply with low maintenance requirements.
3. There is a need to explore more on application areas pertaining to nano-sized ball milled coal fly ash, and behavior (such as, mechanical, thermal, and the like) of fly ash-based composites.

The above gaps have been partially addressed through this experimental investigation.

2.3 Problem definition

The research work describes the development of nanoparticles of thermal power plant waste i.e. fly ash. The particles are fabricated using the top to bottom approach through mechanical ball milling process. The processed fly ash was characterized in order to verify the size of the fly ash and its morphology. Further, aluminium matrix composite was fabricated through the stir casting. The prepared AMC specimens were then tested for tensile and compressive strengths.

2.4 Plan of investigation

The research work was planned to be carried out in the following steps:

1. Statistical analysis of fly ash production, utilization and disposal in India.
2. Study of research carried out on fly ash utilization in the past.
3. Study of method of synthesis of nanoparticles.
4. Preparation of fly ash sample for ball milling process.

5. Conduct of experiments based on the experimental methods conducted in the past.
6. Characterization of the ball milled fly ash sample.
7. Fabrication of aluminium composite reinforced with ball milled nano-fly ash through stir casting process.
8. Conducting mechanical testing to study the tensile and compressive behaviour of the prepared AMC specimen.

CHAPTER 3
THEORY AND EXPERIMENTATION

3.1 An Introduction to Fly Ash

Fly ash, an amorphous structured ferro-alumino-silicate mineral, principally contains micro-sized particles consisting of silicon dioxide (SiO_2), aluminium oxide (Al_2O_3), hematite (Fe_2O_3) with significant oxides of calcium (lime) and magnesium (MgO). These particles are usually light grey in color (depending upon its carbon content), spherical structured and are of alkaline nature [23, 31]. Generally, there are two types of fly ash particles: one is cenosphere-hollow particles (density less than 1 g/cm^3) and the other is precipitator-spherical particles (density in the range of 2-2.5 g/cm^3). The precipitator particles are used for the property improvement such as wear resistance, stiffness, strength, and density whereas cenosphere fly ash particles are used in the preparation of ultra-light composite materials due to low density when compared to metal matrices density. Over the past three and half decades, modern researchers are keen in developing new materials with improved properties for complex applications. Fly ash is an industrial waste from coal power plants and is available in abundance due to its limited use and difficulty in its disposal. It can be used as reinforcement for production of low-cost composite with enhanced mechanical and physical properties for a variety of industrial applications. The physical and chemical properties of fly ash are given in Tables 3.1 and Table 3.2 [4].

Table 3.1 Physical properties of fly ash [4]

Density (g/cm^3)	1.0-2.5
Bulk density (g/cm^3)	1.26
Melting point (°C)	> 1200°C
Modulus (GPa)	143-310
Moisture content, %	2
Particular shape	Regular/irregular
Colour	Gray

Table 3.2 Chemical properties of fly ash [4]

Parameter	SiO_2	Al_2O_3	CaO	MgO	Fe_2O_3	SO_3	Na_2O	K_2O
Wt%	53.1	26.23	4.59	1.10	3.17	0.62	0.21	0.95

3.1.1 Classification of Fly Ash

According to ASTM C-618 [42], two major classes of fly ash are recognized. The two classes are related to the type of coal burned and are designated Class F and Class C in most of the current literature.

1. Class C Fly Ash: It has high cementing abilities, and is formed from the burning of sub-bituminous coal. This kind of fly ash has lime in excess of 20% and also does not need an activator (based on ASTM C 618 standards) for the formation of cementitious compounds. Class C fly ashes are also called high calcium ashes, and they became available for use in concrete industry only in the last 20 years. These are not only pozzolanic in nature but are invariably self-cementitious. When mixed with water, Class C ashes hydrate almost in the same way as Portland cement does.
2. Class F Fly Ash: It is generated from the combustion of bituminous and anthracite coals. These ashes have less than 10% lime content and need an activator (based on ASTM C 618 standards) like Portland cement, quick lime, etc. for the formation of cementitious compounds. Use of Class F fly ash reduces water demands as well as heat of hydration. The concrete made with Class F fly ash exhibits improved resistance to sulphate attack and chloride ion ingress.

Previous research findings and majority of current industry practices indicate that satisfactory and acceptable concrete can be produced with the Class F fly ash replacing 15 to 30% of cement by weight. This class is also used for producing air entrained concrete to improve freeze-thaw durability [15].

3.1.2 Overview of Fly Ash in India

Fly ash is the world's largest generated thermal power plant waste chiefly due to the global dependence on coal-fired power plants [9, 10]. Their physio-chemical properties are highly altered by source and size of coal used, combustion equipment design, process and rate of combustion, coal chemical content and its burning characteristics [11]. About 75% of India's total energy production is coal-based and will remain so for the next few decades.

According to Central Electricity Authority's (CEA) 2017-18 report, the overall utilization of fly ash has escalated swiftly from 1998-99 (9.2 million tons per annum) to 2017-18 (131.8 million tons per annum) [37]. Apprehended from the CEA report, the overall utilization percentage of fly ash contrast to its generation has increased five times in the last two decades. However, in India, a small proportion (of total quantity generated) is being employed for different industrial purposes viz. soil improvement, roadways and pavement constructions, structural components, as an inorganic particulate filler in polymer industry to make polymeric composites. The increased use of coal and its combustion, by-product disposal distress has impacted pressures in all three spheres - air, water and land. Primarily, fly ash disposal in landfill areas hoists a number of concerns regarding its following impact on health and local surroundings [30-37].

About 72% of the Indian population uses coal as the main source of electricity. Power generation in India has increased from 1360 MW in 1947 to about 200 GW in 2012 and is expected to increase to 290 GW in 2047. Annual production of the fly ash in India is therefore also increasing rapidly. From 2015 to 2016, India consumed about 640 million tons of coal (about 25% of the total coal consumption) for power generation. In addition, the quality of coal in India is lower than in most advanced countries, with about 40% ash. This means poorer the quality of coal, more pollutants will be produced, such as PM, NOx.

India's production of the fly ash from 1996 to 2011 is shown in Figure 3.1 below. Production of the fly ash during 2010 and 2011 increased to about 130 million tons, which was about 85.7% higher than during 1996 to 1997. The fly ash utilization rate also increased from 9.63% in 1996 to 1997 to 54.53% in 2010 to 2011, an increase of 466% over a 15 year period. This shows that people are aware of the value of the fly ash utilization with technological advances and increased incomes.

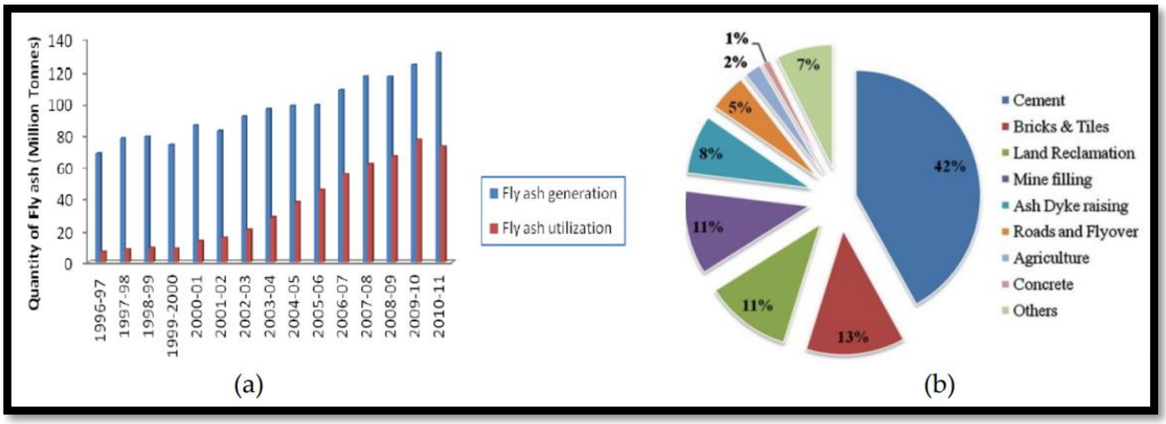

Figure 3.1 Production and utilization of fly ash (a) and uses of fly ash in India (b) [33]

Figure 3.1 shows exemplary uses of the fly ash in India: 42% for cement, 13% for bricks and tiles, 11% for land reclamation, 11% for mine filling, 8% for ash dyke raising, 5% for roads and flyover, 2% for agriculture, 1% for concrete, and 7% for other uses [6]. Given the advantages of the fly ash and the policies for fly ash utilization in India, it is expected that the utilization rate in India will continue to increase in the near future [33]. Figure 3.2 shows some areas of application of the fly ash that are currently being employed.

Another report shows the production and utilization of coal fly ash in India in recent years. The Central Electricity Authority summarized the fly ash generation and utilization from the year 2014 to 2019 as given below in the Table 3.3:

Table 3.3 Year-wise Fly ash production and utilization in India [37]

Description	2014-15	2015-16	2016-17	2017-18	2018-19
Number of Thermal Power Stations	145	151	155	167	195
Installed Capacity (MW)	138915.80	145044.80	145044.80	177070.00	197966.50
Coal consumed (Million Tons)	549.72	536.64	536.4	624.88	667.43
Average Ash Content (%)	33.50	32.94	33.22	31.44	32.52
Fly Ash Generation (Million Tons)	184.14	176.74	169.25	196.44	217.04
Fly Ash Utilization (Million Tons)	102.54	107.77	107.10	131.87	168.40
% Utilization	55.69	60.97	63.28	67.13	77.59

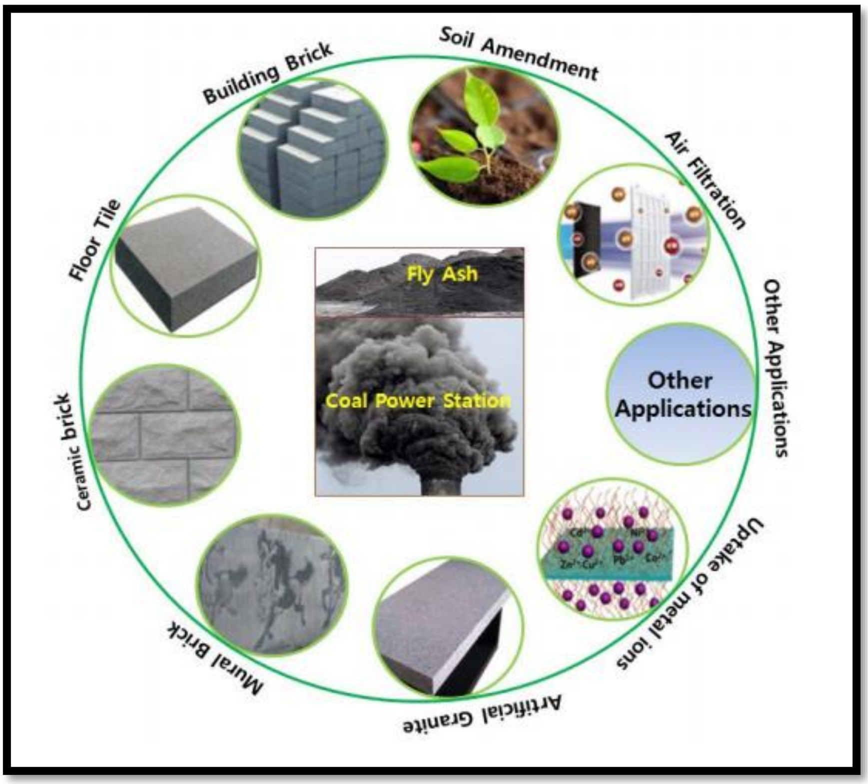

Figure 3.2 Areas of application of the fly ash [21]

3.1.3 Problems associated with Fly Ash

In the past, fly ash produced from coal combustion was simply entrained in flue gases and dispersed into the atmosphere. Fly ash, being a thermal power plant waste is generated in abundance from pulverized coal-burning at power plants. Due to rapid urbanization and industrialization especially in developing countries with high population rate like India, the power consumption is elevated, and this results in operation of new thermal power plants. This created environmental and health concerns that prompted laws that have reduced fly ash emissions to less than 1% of ash produced. Worldwide, more than 65% of fly ash produced from coal power stations is disposed of in landfills and ash ponds. Nowadays, disposal of the fly ash is a major problem, because of the shortage of the landfill sites, increasing costs of the land and strict environmental regulations. In India, landfill covers a vast area and thus, depleting the land for agricultural use. This also creates many environmental hazards if not managed well, and causes soil pollution and ground water pollution [26]. In this regard, Figure 3.3 shows impact of coal fly ash on surroundings, particularly, on water resources.

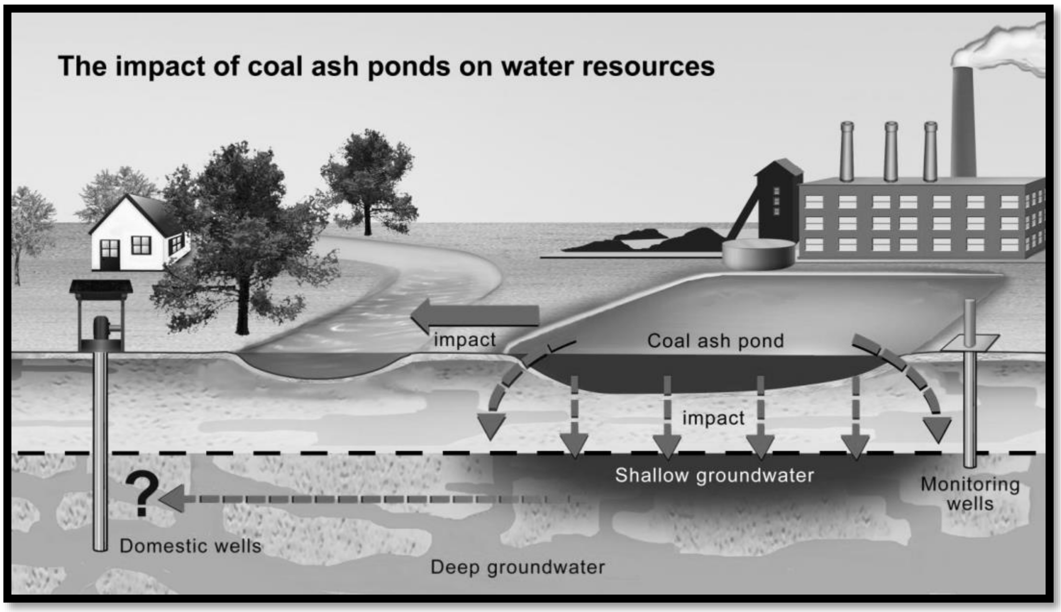

Figure 3.3 Impact of coal fly ash on surroundings [26]

Because of the wet disposal system of the fly ash the metals in it leach and slowly reach aquifer and contaminate water. In India nearly 130 MT of fly ash is spewed out annually. The recycling of fly ash has become an increasing concern in recent years. Environmental benefits of recycling the fly ash include reducing the demand for virgin materials that would need quarrying and cheap substitution for materials such as Portland cement [27].

3.2 Synthesis of Nanoparticles

The nanoparticles are synthesized by various methods that are categorized into bottom-up or top-down approaches and the distinct structured layout of nanoparticle synthesis is shown in Figure 3.4. In essence, four generic routes (namely, approaches) are followed to fabricate nanoparticles, i.e. *wet chemical* (bio-genic, hydrothermal, sol-gel and precipitation processes), *mechanical* (milling, grinding, and mechanical alloying techniques), *form-in-place* (lithography and spray coating processes), and *gas-phase* fabrication (pyrolysis, and high-temperature evaporation techniques). It is sufficiently good in exploring any of the basic routes, because the consequent materials can have significant diversified properties depending upon their chosen fabrication route [2]. Figure 3.5 shows different approaches of synthesis of nanoparticles.

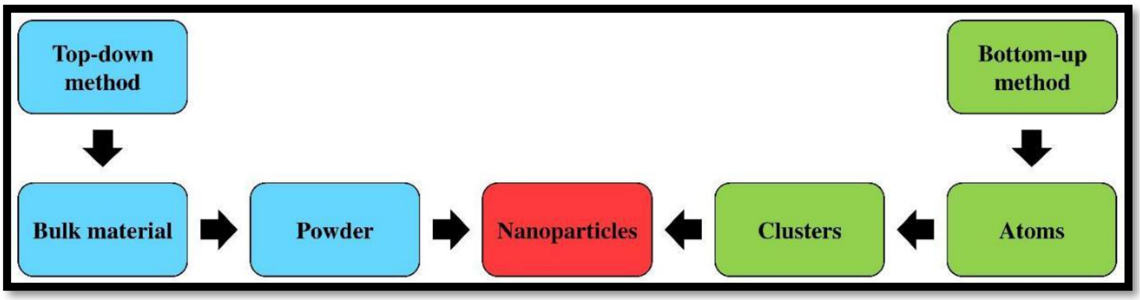

Figure 3.4 Synthesis process of nanoparticles

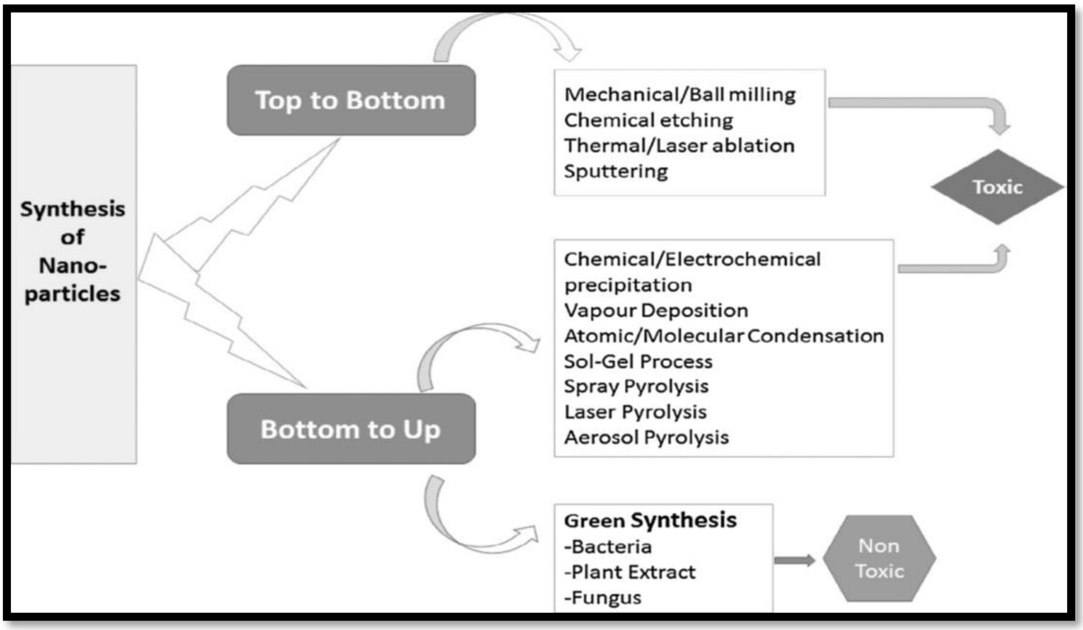

Figure 3.5 Different approaches of synthesis of nanoparticles [1]

Figure 3.6 displays the protocols employed for nanoparticles synthesis and an overview of categories of nanoparticles synthesized from various methods is given in Table 3.4.

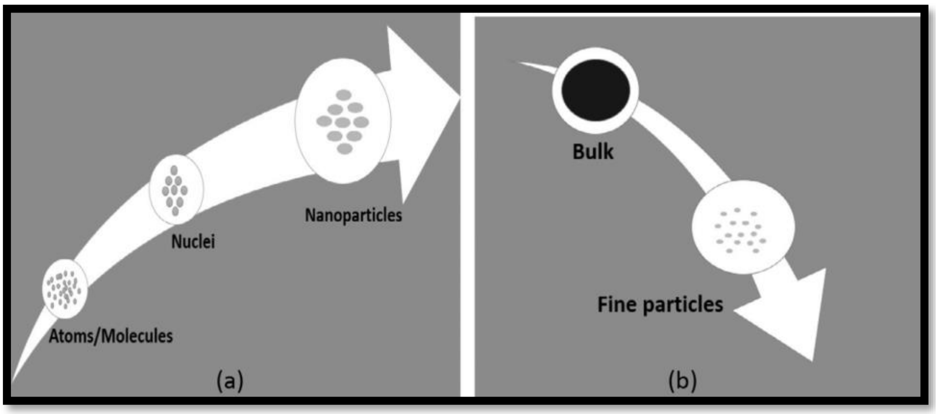

Figure 3.6 Protocols employed for synthesis of nanoparticles (a) bottom to top approach and (b) top to bottom approach [1]

3.2.1 Bottom-up method

Bottom-up or constructive method is the build-up of material from atom to clusters to nanoparticles. Sol-gel, spinning, chemical vapour deposition (CVD), pyrolysis and biosynthesis are the most commonly used bottom-up methods for nanoparticle production. The various bottom-up methods [2] include:

1. Sol-gel: The sol is a colloidal solution of solids suspended in a liquid phase while the gel is a solid macromolecule submerged in a solvent. It is preferred method due to its simplicity. It is a wet-chemical process containing a chemical solution acting as a precursor such as metal oxides and chlorides for an integrated system of discrete particles.
2. Spinning: It is carried out by a spinning disc reactor (SDR) which contains a rotating disc inside a reactor filled with nitrogen or other inert gases where the physical parameters such as temperature can be controlled. The disc is rotated at different speed where the liquid i.e. precursor and water is pumped in. The spinning causes the atoms or molecules to fuse together and is precipitated, collected and dried. Various operating parameters determines the characteristics nanoparticles synthesized from SDR.
3. Chemical vapour deposition (CVD): It is the deposition of a thin film of gaseous reactants onto a substrate. The deposition is carried out in a reaction chamber at ambient temperature by combining gas molecules. A chemical reaction occurs when a heated substrate comes in contact with the combined gas developing a thin film.

Substrate temperature is the influencing factor in CVD which has benefits such as highly pure, uniform, hard and strong nanoparticles.

4. Biosynthesis: In recent decades, biosynthesis has bloomed in developing metallic nanoparticles such as silver, gold, platinum, copper, iron which is finding applications in spheres of biotechnology, bioengineering, textile engineering, catalysis, electronics, sensing, photonics, environmental cleanup, imaging, drug delivery, water treatment, microelectronics, magnetic and antimicrobials [1]. The bio-inspired synthesis of metallic nanoparticles using plant extracts or their parts is simple, easily scaled-up, economical and environmentally benign. The naturally occurring biomolecules in plants have been perceived to play a key role in the formation of nanoparticles with definite shapes and sizes [2]. Biomolecules in the plant extract such as enzymes, proteins, flavonoids, terpenoids and cofactors act as both reducing and capping agents which are responsible for the nanoparticle's synthesis.

3.2.2 Top-down method

Top-down or destructive method is the reduction of a bulk material to nanometric scale particles. Mechanical milling, nanolithography, laser ablation, and thermal decomposition are few nanoparticle synthesis methods which are explained below [2].

1. Mechanical milling: It is used for milling and post annealing of nanoparticles during synthesis where different elements are milled in an inert atmosphere [38-40]. The influencing factors is plastic deformation that leads to particle shape, fracture leads to decrease in particle size and cold-welding leads to increase in particle size.
2. Nanolithography: It is the study of fabricating nanometric scale structures with a minimum of one dimension in the size range of 1 to 100 nm. There are various processes such as optical, electron-beam, multi-photon, nanoimprint and scanning probe lithography. Lithography is the process of printing a required shape or structure on a light sensitive material that selectively removes a portion of material. Its advantage is to produce from a single nanoparticle to a cluster with desired shape and size [2].
3. Laser ablation: The irradiation of a metal submerged in a liquid solution by a laser beam condenses a plasma plume that produces nanoparticles. As LASiS provides a stable synthesis of nanoparticles in organic solvents and water that does not require any stabilizing agent or chemicals it is a ‘green’ process.
4. Thermal decomposition: It is an endothermic chemical decomposition produced by heat that breaks compound’s chemical bonds at a specific decomposition temperature.

Table 3.4 An overview of categories of nanoparticles synthesized from various methods [2]

Category	Method	Nanoparticles
Bottom-up	Sol-gel	Carbon, metal and metal oxide based
	Spinning	Organic polymers
	Chemical Vapour Deposition (CVD)	Carbon and metal based
	Pyrolysis	Carbon and metal oxide based
	Biosynthesis	Organic polymers and metal based
Top-down	Mechanical milling	Metal, oxide and polymer based
	Nanolithography	Metal based
	Laser ablation	Carbon based and metal oxide based
	Sputtering	Metal based
	Thermal decomposition	Carbon and metal oxide based

3.3 Characterization of Nanoparticles

Characterization of nanoparticles is important to understand and control nanoparticles synthesis and assess their applications. It is performed using various techniques such as transmission and scanning electron microscopy (TEM and SEM), atomic force microscopy (AFM), dynamic light scattering (DLS), X-ray photoelectron spectroscopy (XPS), powder X-ray diffractometry (XRD), Fourier transform infrared spectroscopy (FTIR), and UV-Vis spectroscopy. These techniques are used for determination of different parameters such as particle size, shape, crystallinity, fractal dimensions, pore size and surface area. The morphology and particle size could be determined by TEM, SEM and AFM. Moreover, the XRD is used for the determination of crystallinity, while UV-Vis spectroscopy is used to confirm sample formation by showing the plasmon resonance [1]. Furthermore, an Energy-dispersive X-ray spectroscopy (EDX) analysis reveals qualitative and quantitative microanalysis (elemental analysis) of the specimen.

3.4 Ball Milling Technique

Among mechanical methods for nanoparticle synthesis, ball milling, a mechanical technique, has an extensive application in material preparation, activation and synthesis of nanoparticles. Its potential merits encapsulates cost-effectiveness, reliability, low maintenance, ease of working and reproducibility and applicability in wet and dry conditions for materials such as cellulose, chemicals, fibers, polymer compounds and metallic oxides. Its framework consists of a hollow cylindrical shell rotating about its axis (longitudinal axis in case of tumbler mill) and this compartment is partially filled with metal balls usually made of stainless steel, ceramic or rubber [38-40], as shown in Figure 3.7. The efficiency of the process in tumbler mill relies on the diameter of the mill while height of the fall with larger diameters governing the energy generation which is transmitted to the balls. In this mechanical regimen, due to the repetitive action of compressive loads occurring from the ball-powder impacts, a severe plastic deformation is produced. The high concentration of dislocations and defects with sustained interfaces renewal, during milling, enhanced atomic mobility, is conducive to new crystalline and amorphous materials with crystallite structure of Nano-scale [38]. The energy generated because of collision and attrition between the balls and the powder is accountable for its working principle [38]. The position of the ball and their movement inside the vial of a tumbler ball mill (static and dynamic modes) is shown in Figure 3.8 [38]. The exploration of low-energy mill (tumbler mill) over its contemporary high-energy mill (vibratory and planetary mills) is scarce [40].

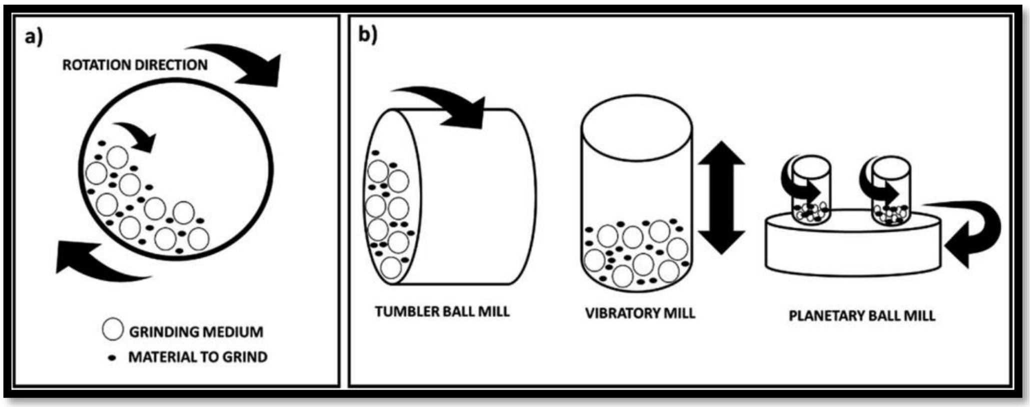

Figure 3.7 Conventional representation of a ball mill (a) and types (b) [38]

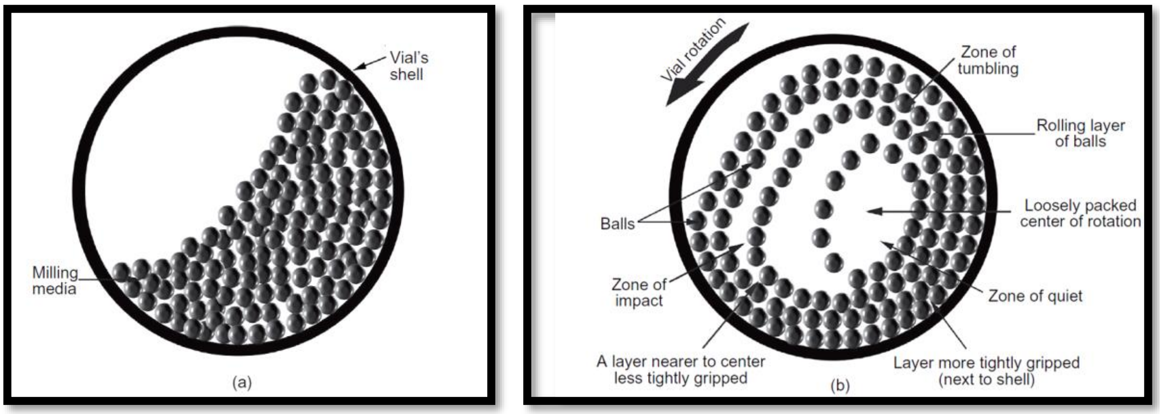

Figure 3.8 Conventional representations show the ball positions and movement inside the vial of a tumbler ball mill at (a) static and (b) dynamic modes [38]

Few demerits are associated with this method i.e. possibility of contamination, formation of nanomaterials with irregular shape, noise, long milling and cleaning times.

3.5 Stir Casting Method

Metal matrix composite are being manufactured with the help of a stir casting process which is a liquid state method. It is a process of melting a material with continuous stirring and immediately pouring the melt into a preformed cavity, then cooling it and allowing it to solidify. During this process, the particles often tend to agglomerate, which can be only dissolved by vigorous stirring at high temperature. The stir casting set up (Figure 3.9) comprises of a furnace, crucible and a rotor attached to the motor. A stirrer is employed to agitate the molten metal matrix. The stirrer is generally made up of a material which can withstand high melting temperature than the matrix temperature.

Typically, a graphite stirrer or a stainless steel stirrer is used in the stir casting. It consist of two components i.e. cylindrical rod and impeller. It is held in vertical position where one end of rod is connected to impeller and other end is connected to shaft of the motor. The resultant molten metal is then poured in die for casting. The stir casting is suitable for manufacturing composites with up to 30% volume fractions of reinforcement. A major concern associated with the stir casting is segregation of reinforcement particles due to various process parameters (wetting condition of metal particles, relative density, setting velocity) and material properties which result in the non-homogeneous metal distribution. The distribution of particle in the molten metal matrix is also affected by velocity of the stirrer, angle of the stirrer, etc.

Initially, the matrix metal is heated above its liquid temperature so that it is transformed into molten state. This is followed by cooling it to temperature between liquid and solidus state i.e. it is in a semi-solid state. Then preheated reinforcement particles are added to molten matrix and again heated to fully liquid state so that they are mixed thoroughly [4].

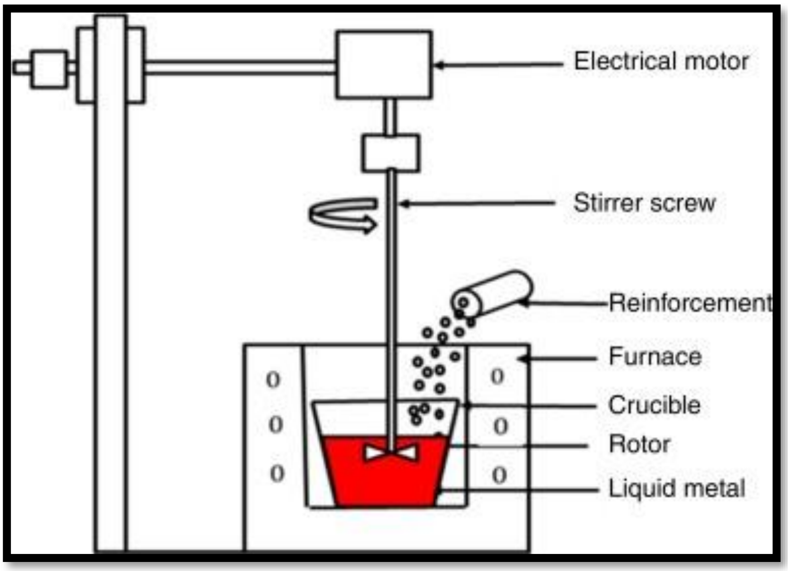

Figure 3.9 Line diagram of the stir casting process [28]

3.6 Experimental Method

3.6.1 Sample Preparation of Fly ash for Ball Milling

The fly ash was obtained from thermal power station, Panipat, India. Fresh fly ash was washed in distilled water and the carbon that creamed up during washing was removed. It was then dried at 100ºC for 6 hours to remove water content. Dried fly ash was sieved using ASTM standard sieve. The fly ash fractions that passed through mesh nos. 70 and 140, but got retained on mesh no. 200 were collected and magnetic separation was carried out manually to remove the magnetic impurities. Then this fly ash was taken for ball milling to ball milling set up (Figure 3.10).

The following milling conditions were maintained: the ball mill was loaded with balls at 10:1 ratio to the fly ash; milling chamber and balls were made of stainless steel; the ball diameter was 8 mm and weight of 16.5 g. Ball milling was carried out in the presence of powdered sodium lauryl sulphate (SLS) as the surfactant to avoid agglomeration of the ceramic particles in the fly ash, at a ratio of 100:1 of the fly ash to surfactant. To avoid the generation of high temperature, the milling time was set to 120-min working with 30-min breaks. The sample of the milled powder was taken out after a total of 1200 min and dried in a hot air oven at 80ºC for 2hrs prior taking for characterization.

The ASTM standard sieve analysis is shown in Figure 3.11, wherein the fly ash fractions are passed through preselected meshes.

Figure 3.10 Ball mill machine set-up

Figure 3.11 ASTM standard sieve analysis

3.6.2 Fabrication of Aluminum-based Nanocomposite

The fabrication of aluminum-based nanocomposite of composites was done by the stir-casting method. Aluminum alloy (Al 4104) was taken as base matrix metal, and the ball milled fly ash along with titanium dioxide (TiO_2) were taken as reinforcements. The ball milled fly ash was taken in 0% and 10% mass fraction (namely, weight percentage), while TiO2 was taken in 0%, and 7% on mass fraction. The Al 4104 alloy ingots were kept in crucible and melted in furnace (attached with the stir casting setup) at 1200˚C. The crucible was kept in the furnace till the aluminum completely melts. Thereafter, calculated amount of the preheated reinforcements of ball milled fly ash and/or TiO_2 were added slowly into the melt. The mixture was stirred continuously. The stirring action was carried out for about 12 minutes. Further, the mixed melt was poured to the sand mold of 22 mm diameter and 300 mm length (for tensile and compressive specimen). The molten metal was allowed to solidify, and the prepared casting was removed from the molds. The casted composites were cut into required sizes of pieces and then machining operations were carried out in order to prepare the specimens for the tensile and compressive tests.

3.6.3 Preparation of Specimens for Mechanical Testing

A conventional lathe machine, a vertical milling machine and a bench grinder machine were employed to prepare specimens for the mechanical testing. The specimens prepared were tensile test specimens and compressive test specimens with dimensions as indicated in Figures 3.12 and 3.13 respectively. Computer-aided designs (CADs) for the tensile test specimens and the compressive test specimens were also made using SOLIDWORKS 2013, as shown in Figures 3.14 and 3.15, respectively.

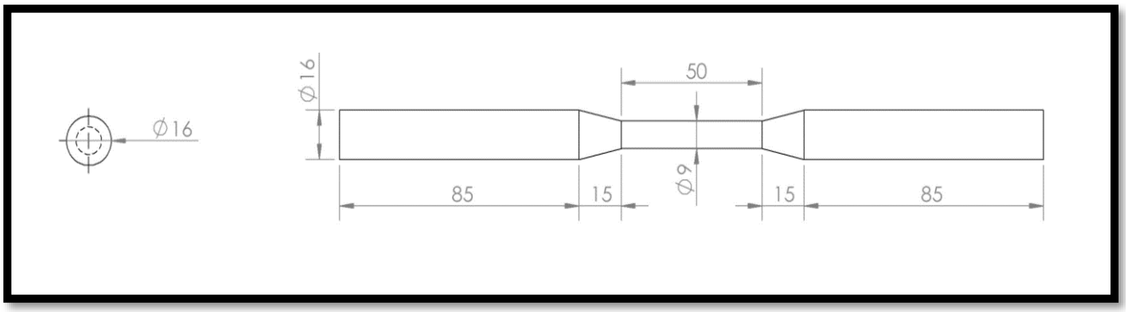

Figure 3.12 Dimensions of tensile test specimen

Figure 3.13 Dimension of compression test specimen

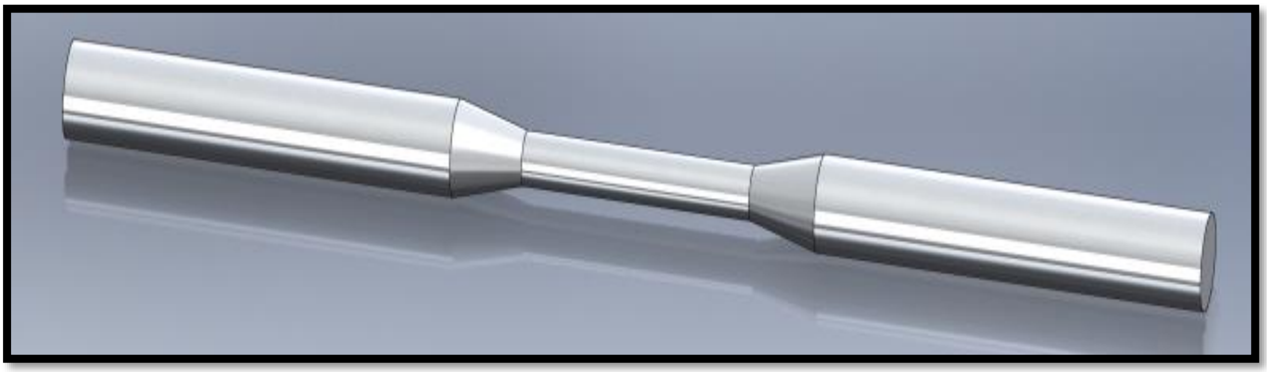

Figure 3.14 CAD for tensile test specimen

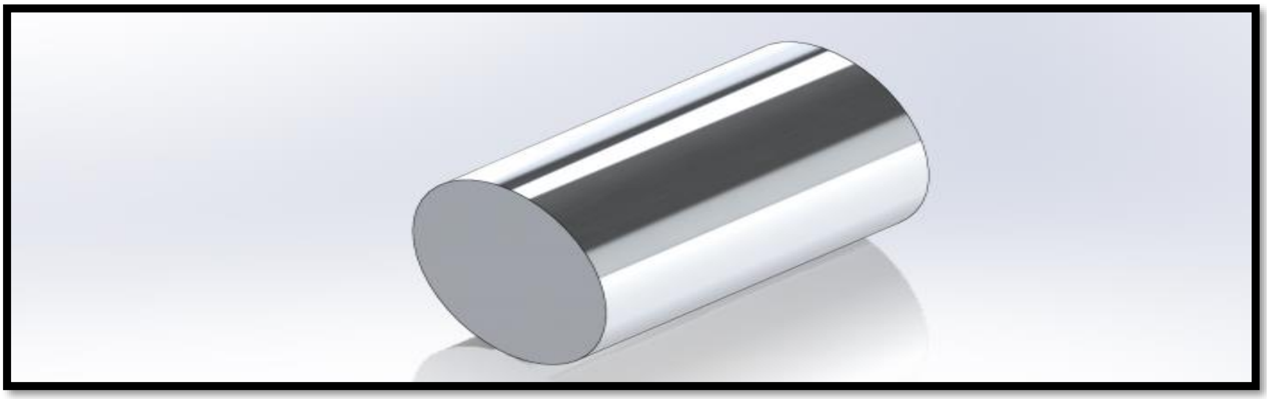

Figure 3.15 CAD of compression test specimen

3.6.3.1 Fabrication steps involved in the tensile test specimen preparation

Initially, the cylindrical shaped workpiece (of dimensions: D = 22 mm, and L = 260 mm) was sectioned from the casted composite using the vertical milling machine. Then, using the conventional lathe, facing operation was performed on both ends and the length of the piece was reduced to 250 mm. Further, turning operation was performed and the diameter of the workpiece was reduced to 16 mm. Then, a gauge length 50 mm was made from the center of the specimen and diameter was maintained as 9 mm. Finally, using the conventional lathe, taper turning operation was performed to produce the desired shape of the tensile test specimen. Figure 3.16 shows the preparation of the tensile test specimen on a conventional lathe machine.

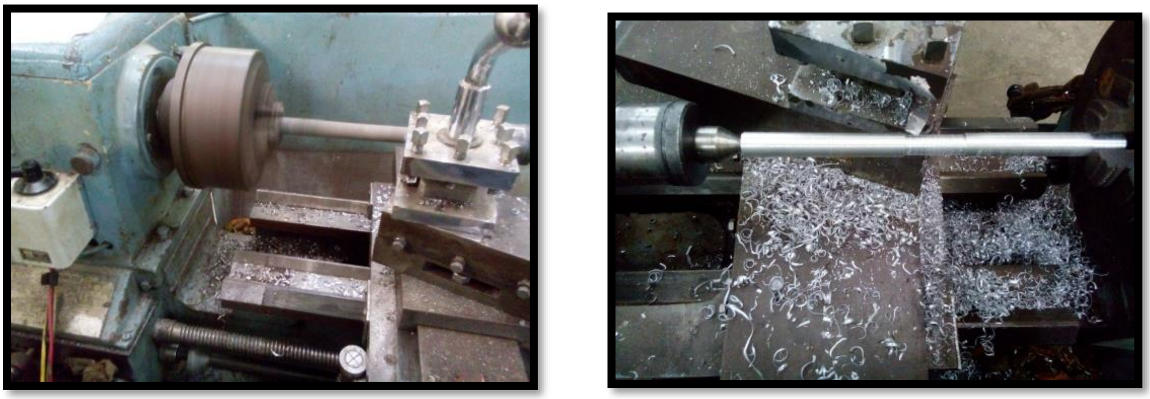

Figure 3.16 Tensile test specimen preparation on a conventional lathe machine

3.6.3.2 Fabrication steps involved in the compression test specimen preparation

Initially, the cylindrical shape workpiece (dimensions: D = 22 mm, and L = 30 mm) was sectioned from the casted composite using the vertical milling machine. Then, using the conventional lathe, facing operation was performed on both ends and the length of the work piece was reduced to 25 mm. Further, turning operation was performed and the final diameter (15 mm) was achieved. Figure 3.17 shows the preparation of the compression test specimen on a conventional lathe machine.

Figure 3.17 Compression test specimen preparation on a conventional lathe machine

CHAPTER 4
RESULTS AND DISCUSSION

4.1 Study of Chemical Composition of Fly ash: EDX Analysis

There are two genre of coal fly ash that have been established and classified by the authority of American Society of Testing Materials (ASTM C618 2000) i.e. Class F and Class C fly ash. The class F fly ash is yielded from combustion of anthracite or bituminous coal, while the class C fly ash is produced from combustion of lignite or sub-bituminous coal. This taxonomy is based on the dissimilitude of concentrations of SiO_2, Al_2O_3, and Fe_2O_3 and also loss of ignition in the produced ash. For the classes C and F, the cumulative percentage of aforementioned oxides should be greater than 50% and 70% respectively, and loss of ignition should be less than 6% and 12% respectively. In addition, high lime content ranging 10-40% has been observed for the class C fly ash, while the class F fly ash has less than 10% lime content [41]. The chemical composition of the procured fly ash was analyzed by EDX analysis which is displayed in Table 4.1. From the calculations reported from the EDX analysis, the composition of the procured fly ash altogether derived for this experimentation constitutes major segments of SiO_2, Al_2O_3, and Fe_2O_3, which adds up to 94.98%. Furthermore, the percentage of CaO (3.14%) is less than that of Fe_2O_3 (8.78%). This observation divulges that the procured fly ash sample belongs to class F which agrees well with findings of K. T. Paul et al. [42].

Table 4.1 Chemical composition of raw coal fly ash (wt %)

Element composition		Oxide composition	
Component	Content	Component	Content
Silicon	50.23	Silicon oxide (SiO_2)	52.36
Aluminum	33.21	Aluminum oxide (Al_2O_3)	33.84
Iron	8.15	Iron oxide (Fe_2O_3)	8.78
Calcium	4.13	Calcium oxide (CaO)	3.14
Titanium	1.02	Titanium oxide (TiO_2)	1.32
Magnesium	1.30	Magnesium oxide (MgO)	0.56

4.2 Mineralogical Study of Fly ash: XRD Analysis

XRD analysis of the fly ash is carried to identify various phases involved due to the presence of high amorphousness along with peak-overlaps and weak peak intensities [43].

X-ray diffractogram of the raw coal fly ash is given in Figure 4.1 representing peak intensities of major fly ash constituents. Diffraction pattern study of raw fly ash shows the existence of numerous crystalline peaks despite exhibiting lower degree of crystallinity of fly ash and amorphous halo patterns stretching approximately from 15° to 45° (2θ) inferring the presence of non-crystalline glassy solids. The substantial crystalline phases of the fly ash samples were majorly recognized as quartz, hematite and mullite. This was coherent with the outputs of the EDX analysis conducted. Mullite (alumino-silicate) exhibits peaks at 16.32°, 25.98°, 30.99° and 40.72° at 2θ values. The quartz (silicon dioxide) shows peaks at 20.46°, 26.32°, 35.94°, 41.93°, 49.60°, 54.69° and 61.38° at 2θ values and hematite (ferric oxide) at 33.06°, 36.40° and 68.10° at 2θ values.

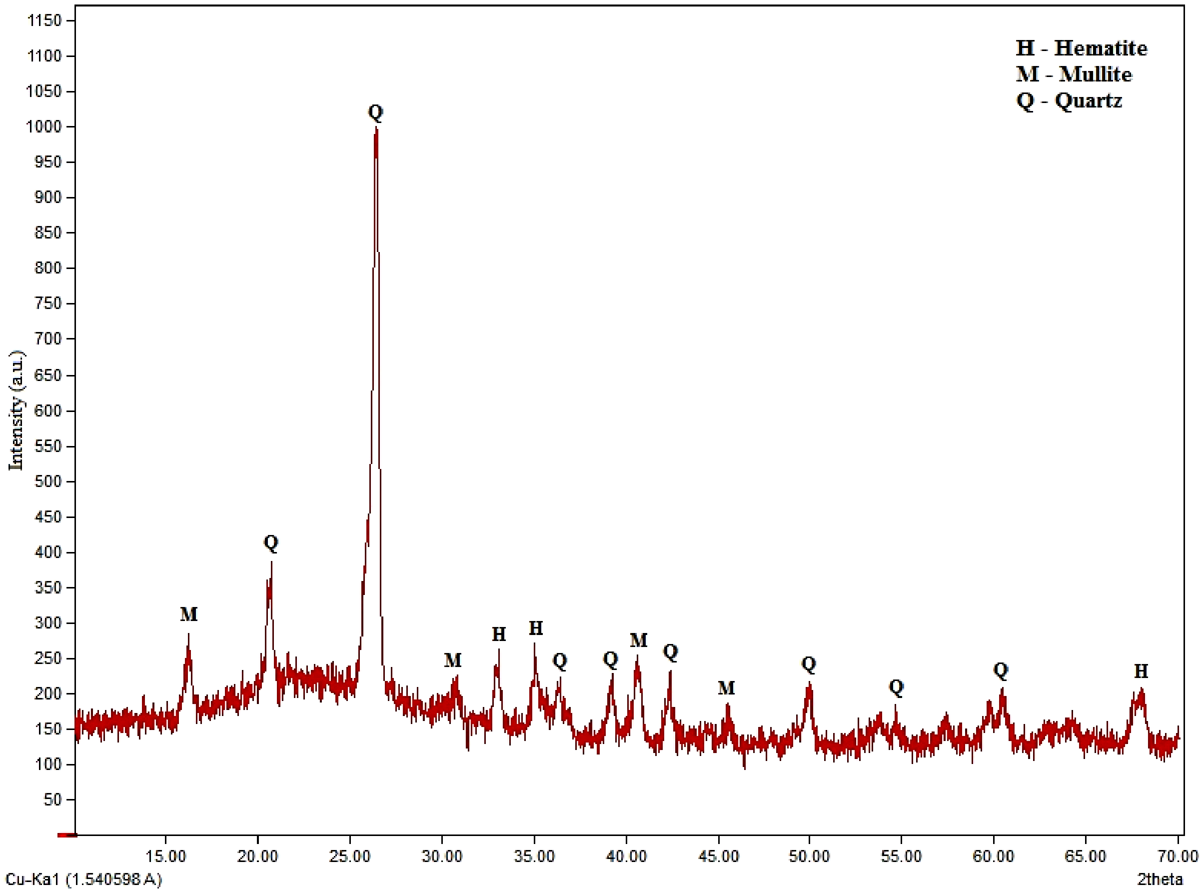

Figure 4.1 X-ray diffraction pattern of raw fly ash

Figure 4.2 depicts comparison of the X-ray diffractograms of raw fly ash and ball milled fly ash after 600 min and 1200 min of milling. A steady drop in the crystallite size is observed, mostly affecting the crystallite size of quartz phase. It is clearly observed that peak intensity of quartz phase (also other original crystallinites present) is significantly diminished and the peaks broaden with increase in the duration of milling. These peculiar observations have also been reported by Hui Li et al. [44] and Yong-Sik C. et al. [45]. The reason behind this can be elucidated to the smaller particle size and enhanced amorphous domain post ball-milling process.

The average crystallite size of the XRD peak was determined by the full width at half maximum (FWHM) using Debye-Scherrer's equation [46].

$$d = \frac{K\lambda}{B\cos(\theta)}$$

where 'd' represents the average crystallite domain size perpendicular to the reflecting planes, 'K' denotes crystallite shape constant (0.89 taken for this study), 'λ' is the wavelength of Cu-Kα radiation (typically, λ = 0.15406 nm), 'B' stands for the half width of the diffraction peak (FWHM) and 'θ' represents the incident angle (26.5569° from XRD result) of x-ray surface.

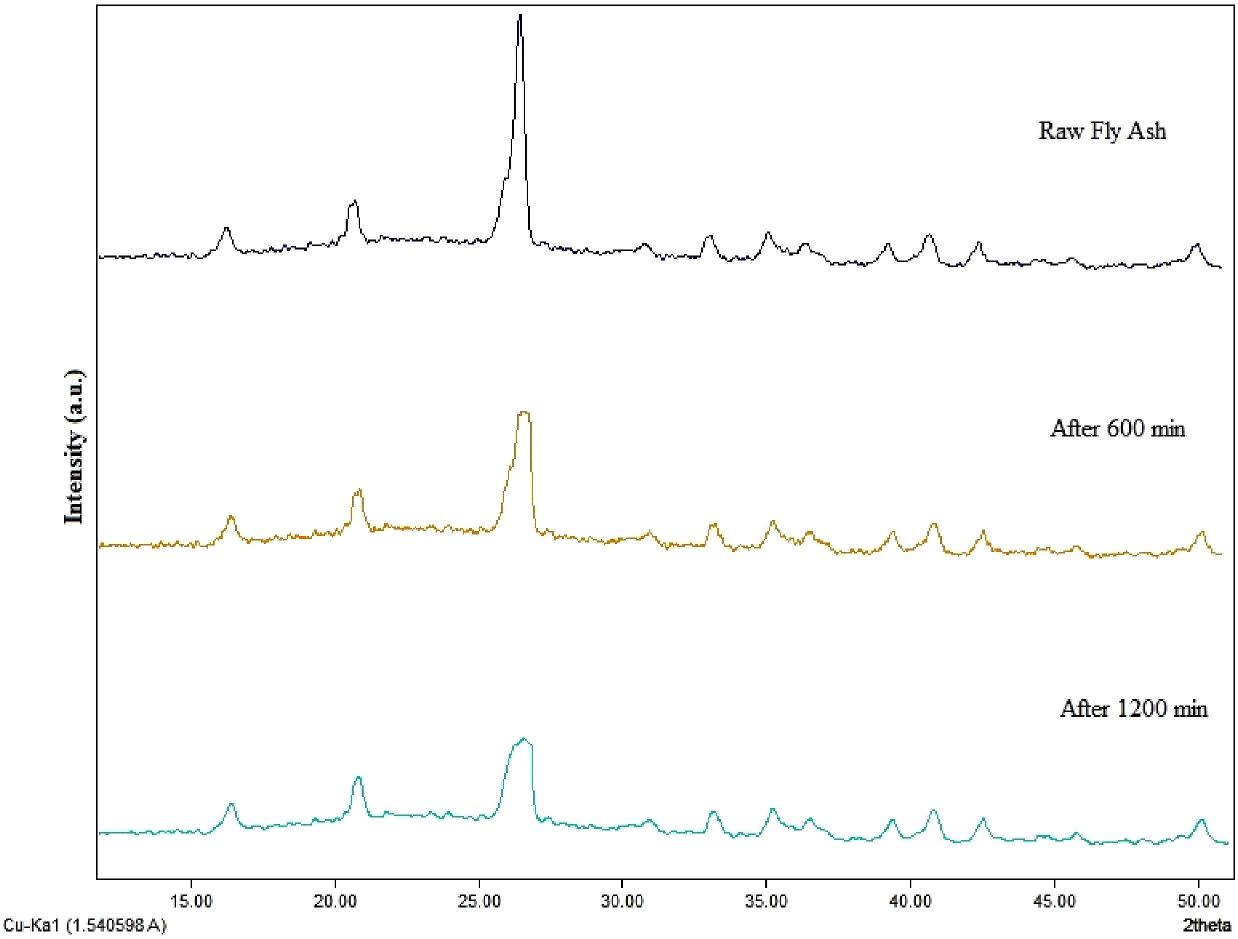

Figure 4.2 X-ray diffractograms of raw and milled fly ash at different times.

After the ball-milling process, the crystallite size of the quartz phase decreased to 22.38 nm (average crystallite domain size ~30 nm), and this change indicative of an increase in amorphousness of fly ash that would help in achieving better compatibility and significantly higher reactivity with various polymeric matrices. During a start of the ball milling process, the sharp decrease in crystallite size is achieved through dispersion and it has been observed that with a higher ball-mill time, the size might have increased due to the effect of agglomeration [47]. Beside the point, the XRD analysis also reported the calculated density as 2.553 g/cm^3 and trigonal crystal system for quartz phase, thereby making the ball milled fly ash suitable as reinforcement filler for light weight density composites.

4.3 Morphology Study of Fly ash: SEM Analysis

The microstructure and surface topography of the raw fly ash and the ball milled fly ash were investigated using electron microscope. Figure 4.3 (a) shows the SEM image of raw fly ash. Mostly, the particles of raw fly ash possess a spherical shape and have a mean diameter greater than 5 microns. The conditions of coal combustion such as kindling temperature and rate of cooling administer the morphology of the fly ash. In power plants using pulverized coal fired boilers, the operating temperature of furnace seldom exceeds 1500°C. During combustion, inorganic compounds present in coal behave like liquids before cooling down. At these elevated temperatures, coal-minerals may oxidize, decompose, fuse, dissolve or aggravate to give diverse morphologies to the fly ash particles.

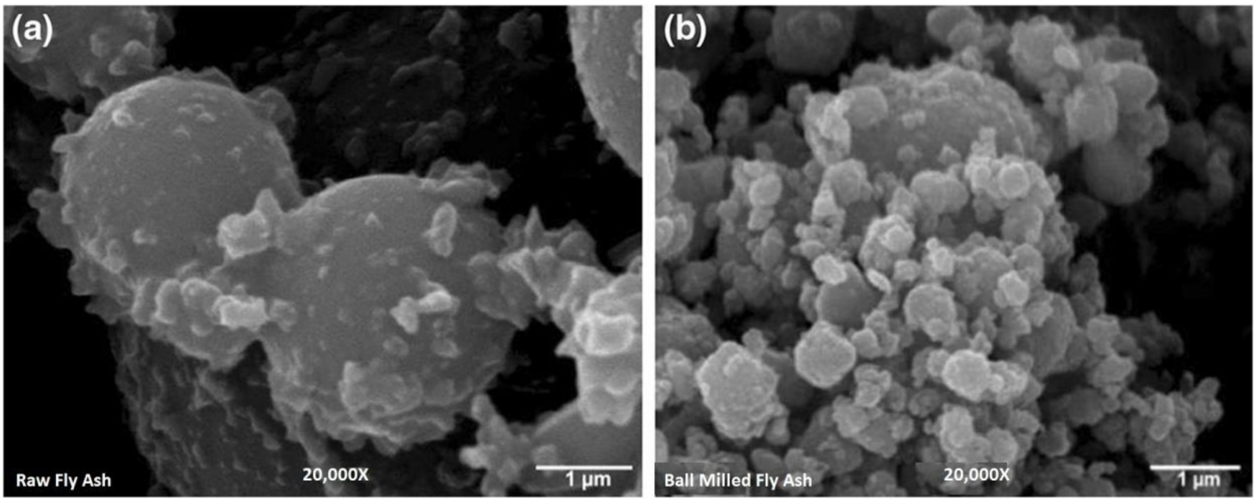

Figure 4.3 SEM photomicrographs of (a) raw and (b) ball milled fly ash

Figure 4.3 (b) shows the photomicrograph of ball milled fly ash after 1200 min of milling at the magnification of 20kX. The original structure of raw fly ash has been destroyed and this tear down has taken the edge off its average particle size. The scope of breakdown of fly ash structure is more pronounced when the milling time increases and as a result, produces finer particle. Thus, the nano-structured materials are anticipated to prevail in the fly ash after ball milling process. Also the surface topography of fly ash indicates that its transformation from a smooth texture to a rough and asymmetrical one. The increase in surface roughness of fly ash also raises its surface energy [41]. It can be theorized that during the ball milling process, the existing glassy lustre of raw fly ash particles on the outside may be abraded and the kernel of the crystal may be condemned after milling for 1200 min.

4.4 Composite Composition of AMC specimens

Cylindrical rods measuring 250 mm in length and 16 mm in diameter were cast for 3 different cases as shown in Table 4.2. The rods after stir casting in the un-machined condition weighed between 350g-450g as shown in Figure 4.4.

Table 4.2 Composite Composition of AMC specimens (in weight percentage)

Specimen	TiO_2	FLY ASH	ALUMINIUM
ROD1	0%	10%	90%
ROD2	7%	0%	93%
ROD3	7%	10%	83%

ROD 1

ROD 2

ROD 3

Figure 4.4 Un-machined Stir-casted Cylindrical Rods

4.5 Mechanical Testing of Prepared AMC Specimens

The prepared AMC specimens were taken for mechanical testing viz. tensile test and compression test. These tests were conducted in order to check the ductile behavior of aluminium composite.

4.5.1 Tensile Test of AMC Specimens

The tensile strengths of AMC test specimen rods 1, 2 and 3 are shown in Table 4.3. The tensile strength of pure aluminium Al-4104 is 110 MPa as per the standard. Experiments were performed by casting the base material i.e. Al 4104 with titanium dioxide (TiO_2) and fly ash as reinforcements individually and collectively. When only fly ash was added to the base metal, the tensile strength showed an increase of approximately 3% but at the expense of ductility. To maintain the ductility, another reinforcement was selected i.e. titanium dioxide. It was added individually to base metal and tested for the tensile strength. An increase of 7.3% was recorded in the tensile strength with minimal change in ductility. Hence, another composite was fabricated consisting of both fly ash and titanium dioxide as reinforcements. The strength was achieved to be in accordance to the strength of base metal while maintaining the ductility of the composite. The weight percentage of the reinforcements was selected after an extensive review of previously conducted research.

Table 4.3 Tensile Strength of Specimens

Specimen	**Composition**	**Tensile Strength (N/mm^2)**
Rod 1	0%TiO_2 + 10%Fly ash + 90%Aluminium	113.05
Rod 2	7% TiO_2 + 0%Fly ash + 93%Aluminium	118.11
Rod 3	7% TiO_2 + 10%Fly ash + 83%Aluminium	111.03

4.5.2 Compression Test of AMC Specimens

A compressive strength analysis was conducted for the prepared AMC specimens as they showed a change in material behavior owing to the addition of reinforcements. A brittle behavior was observed due to the presence of fly ash. Also, previous literature related to fly ash being used in construction material, showed the compressive strength as the key factor. Hence, this compression test was important to identify the brittle nature of the composite. The results obtained are depicted in Table 4.4. It clearly indicates that when adding titanium dioxide and fly ash collectively, an optimum value of compressive strength can be achieved.

Table 4.4 Compressive Strength of Specimens

Specimen	Composition	Compressive Strength (N/mm^2)
Rod 1	0%TiO_2 + 10%Fly ash + 90%Aluminium	300.02
Rod 2	7% TiO_2 + 0%Fly ash + 93%Aluminium	266.52
Rod 3	7% TiO_2 + 10%Fly ash + 83%Aluminium	277.50

Another method to identify the brittle nature is the value obtained from ratio of tensile strength to compressive strength. The lower the ratio, the more brittle is the material. Table 4.5 shows the ratio of tensile strength to compressive strength for all the specimens.

Table 4.5 Brittle Nature based on ratio of Tensile Strength and Compressive Strength

Specimen	Composition	Tensile Strength (N/mm^2)	Compressive Strength (N/mm^2)	Value of Ratio
Rod 1	0%TiO_2 + 10%Fly ash + 90%Aluminium	113.05	300.02	0.376
Rod 2	7% TiO_2 + 0%Fly ash + 93%Aluminium	118.11	266.52	0.443
Rod 3	7% TiO_2 + 10%Fly ash + 83%Aluminium	111.03	277.50	0.400

The above mechanical testing shows that brittle behavior can be controlled by addition of titanium dioxide as a reinforcement while using the fly ash as a reinforcement in base metal. Hence, it can be inferred that the fly ash-based composites can be used in areas where aluminum composites are being used.

CHAPTER 5
CONCLUSIONS

5.1 Conclusions

After accomplishment of this experimental investigation, followings conclusions were drawn:

1. The literature study on the fly ash, the aim of conversion of significant large particles of the raw fly ash to fly ash nano particles, and the fabrication of aluminium-based nanocomposite were accomplished successfully.
2. EDX analysis revealed that the fly ash used for this study belongs to class F.
3. After 12 hours of milling (low energy ball milling) of procured fly ash, the crystallite size (in XRD analysis) of quartz phase present in the procured fly ash was reduced from ~46 nm to 22.38 nm.
4. As evidenced from SEM studies, the spherical shape and smooth surface texture of the procured fly ash was changed into an irregular shape, a rough and a reactive surface by the ball milling process implying more amorphousness and likely a higher reactivity of the ball milled fly ash toward more applicable utilization.
5. The completion of the study showed that the low energy mills may lead to an increase in the required milling time for a complete mechanical alloying process and produce homogeneous and uniform powders. In addition to this, using the low energy mills would be inexpensive than those of the high-energy mills and can be self-made with low cost investment with low maintenance requirements.
6. The fabrication of aluminium-based nanocomposite reinforced with ball milled coal fly ash was done successfully via the stir casting route.
7. The mechanical testing of nanocomposite specimens inferred that with appropriate weight percentage of the fly ash and the titanium dioxide, the tensile strength and the compressive strength can be enhanced optimally without compromising basic nature or behavior of the base material.

5.2 Future scope

Researchers are nowadays experimenting the utilization of thermal power plant waste i.e. fly ash for strengthening of materials and development of new materials, usually composites. Fly ash plays a vital role in enhancing the mechanical properties such as tensile strength, compression strength and hardness of the metal. In today's modern materials sector, a light weight composite material with higher hardness, higher strength, and improved stiffness is being demanded in every sector. The obtained nano-sized fly ash can be used as a reinforcement which showed improvement in physical and mechanical properties in thus formed composites or hybrid composites. As evidenced, more amorphousness and a higher reactivity of the ball milled fly ash proves its novelty towards more applicable utilization at comparatively lower cost.

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