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Micro-structural examination of composite nano-material developed in designed and fabricated cold static compaction chamber

Amir Hussain Idrisi¹, Gaurang Deep², Ayush Singh³

^{1, 2}Assistant professor, Department of Mechanical Engineering, SRMS CET, Bareilly

³Assistant professor, Department of Mechanical Engineering, Amity University, Lucknow

Abstract: Nanotechnology may be regularly exceptionally expansive term, including fields about science similarly as different Concerning illustration surface science, organic chemistry, atomic biology, semiconductor physics, micro creation and so on. The associated research also provisions would just as diverse, going starting with extensions of traditional gadget material science should totally new methodologies built upon atomic self-assembly, starting with Creating new materials with extents on the nanoscale will immediate control at whatever intricate framework. In the Different requesting fields from engineering and medical such as aerospace, defence, automobiles, electronics, materials, chemistry, energy, environment, majority of the data & communication, customer products and bio-technology, its zone about provisions turning into wider and wider. An extensive variety from claiming research need been finished on the transforming routines furthermore material properties about Al-Al₂O₃ nano-composites. Recently, a few methods in ultra nationalistic helped casting, nano-sintering, powder metallurgy, high energy ball milling, friction stir processing are being connected for those development for Al- Al₂O₃ nano composites. In the exhibit fill Cold Isostatic Compaction Chamber used for compaction of metal, alloys and composite powders. Al-Al₂O₃ nano composite with 1 to 3 weight % of nano Al₂O₃ particles were fabricated through powder metallurgy process using Cold Isostatic Compaction Chamber. The micro-structural analysis is carried out on the specimen and results are recorded.

Keywords: Aluminium, Al₂O₃, microstructure, SEM, composite material.

I. INTRODUCTION

A. Composite materials

Composite materials are made from two or a greater amount constituent materials for essentially diverse physical or concoction properties, that when combined, generate a material with qualities unique in relation to those distinctive segments. The distinctive segments remain separate Furthermore unique inside the completed structure. When those grid will be an metal, the composite is termed a metal-matrix composite (MMC). Previously, MMCs, generally those reinforcements need aid in the manifestation for particles, whiskers, short fibers, or continuous fibers.

1) Types of Composite Materials

Typical engineered composite materials include:

Composite building materials such as cements, concrete etc.

Reinforced plastics such as fiber-reinforced polymer (FRP)

Metal Matrix Composites

Ceramic Matrix Composites

Composites likewise use metal fibres reinforcing other metals, concerning illustration done metal matrix composites (MMC). Ceramic matrix composites are constructed principally to crack toughness, not for quality illustration for CMCs would cement (ceramic and metal), concrete and so on. Organic matrix/ceramic aggravator composites incorporate black-top concrete, mastic asphalt, mastic roller hybrid, dental composite, syntactic froth and mothball from claiming pearl. Chobham shield may be an unique kind about composite shield utilized within military provisions. At the a standout amongst a greater amount of the extent of the support will be for 1 will 100 nm measure range, that point it will be arranged Similarly as Nanocomposite.

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B. Nanocomposite

An nanocomposite is a multiphase strong material the place a standout amongst those stages need one, two or three extents for short of what 100 nanometers (nm) size, alternately structures hosting nano-scale repeatable distances the middle of those separate stages that make up those material [1]. In the broadest feeling this definition can incorporate porous media, colloids, gels Also copolymers, in any case is a greater amount generally made to mean the strong blending of a heft grid and nano-dimensional phase(s) varying to properties because of dissimilarities to structure and properties. Those mechanical, electrical, thermal, optical, electrochemical, reactant properties of the nanocomposite will differ markedly from that of the part materials. Extent breaking points for these impacts have beenproposed [2] <5 nm for reactant activity, <20 nm for making an difficult attractive material soft, <50 nm for refractive file changes, and <100 nm for accomplishing super paramagnetism, mechanical fortifying alternately confining matrix disengagement development.

Nanocomposites would discovered Previously, nature, for illustration in the structure of the abalone shell also bone. The utilization about nanoparticle rich materials in length predates those seeing of the physical and compound nature for these materials. Jose-Yacaman et al. [3] investigated those root of the profundity of shade and the safety with acids and bio-corrosion from claiming maya blue paint, attributing it should a nanoparticle system. From the mid-1950s nanoscale organo-clays have been used to control stream about polymer results (For instance as paint viscosifiers) or those constitution of gels (e.g. Likewise a thickening substance on cosmetics, keeping those arrangements in homogeneous form). Eventually by 1970s polymer/clay composites were those themes for textbooks [4], despite those terms "nanocomposites" might have been not in normal use.

In mechanical terms, nanocomposites contrast from routine composite materials because of the exceptionally secondary surface range to volume proportion of the reinforcing period or its exceptionally secondary perspective proportion. The reinforcing material could a chance to be produced dependent upon about particles (e.g. Minerals), sheets (e.g. Exfoliated dirt stacks) alternately fibres (e.g. Carbon nano tubes or electro spun fibres). The zone of the interface the middle of those grid and support phase(s) may be commonly a request of extent more stupendous over to customary composite materials. The grid material properties are essentially influenced in the region of the support. Ajayan et al. [1] noted that for polymer nanocomposites, properties identified with local chemistry, level of thermo situated cure, polymer chain mobility, polymer chain conformation, degree for polymer chain requesting or crystallinity can constantly on shift essentially and more ceaselessly from the interface for the support under those bulk of the matrix.

1) Types of Nanocomposites

Ceramic Matrix Nanocomposites (CMNC)

Metal Matrix Nanocomposites (MMNC)

Polymer Matrix Nanocomposites (PMNC)

- a) Ceramic grid Nanocomposites (CMNC) In this group of composites the principle portion of the volume is accompanied by a ceramic, that is a synthetic compound from the aggregation about oxides, nitrides, borides, silicides and so on. To The greater part cases, ceramic-matrix nanocomposites encompassa metal similarly as the second part. Ideally both components, the metallic particular case and the ceramic one, are finely scattered for one another in place will elicit the specific nanoscopic properties. Nanocomposites from these combinations were showed to enhancing their optical, electrical and magnetic properties additionally tribological, corrosion-resistance and different protective properties. Ceramic matrix Nanocomposites incorporate $\text{Al}_2\text{O}_3/\text{SiO}_2$, SiO_2/Ni , $\text{Al}_2\text{O}_3/\text{TiO}_2$, $\text{Al}_2\text{O}_3/\text{SiC}$, $\text{Al}_2\text{O}_3/\text{CNT}$ etc.
- b) Metal Matrix nanocomposites (MMNC) refers to materials comprising of a ductile metal alternately alloy matrix in which exactly nanosized support material is embedded. These materials consolidate metal and ceramic features, i.e. ductility and toughness with high strength also modulus. Thus, metal matrix nanocomposites are suitability for generation of materials for high strength quality for shear/compression procedures also secondary administration temperature abilities. They indicate an uncommon possibility to requisition in numerous areas, for example, such that aerospace, auto businesses furthermore other metal matrix Nanocomposites incorporate $\text{Al}/\text{Al}_2\text{O}_3$, Al/SiC , $\text{Fe-Cr}/\text{Al}_2\text{O}_3$, $\text{Ni}/\text{Al}_2\text{O}_3$, Co/Cr , Fe/MgO , Al/CNT , Mg/CNT and so on.
- c) Polymer Matrix Nanocomposites (PMNC):- In the simplest case, suitably including nano-particulates with a polymer grid might upgrade its performance, often previously, exact emotional degree, toward basically capitalizing on the nature with more

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properties of the nanoscale filler (these materials are exceptionally depicted by the term nanofilled polymer composites). This methodology may be especially powerful done yielding high performance composites, The point when handy scattering of the filler will be attained and the properties of the nanoscale filler are significantly distinctive or superior to the individuals of the matrix, to example, reinforcing a polymer grid toward a significant part stiffer nanoparticles from claiming ceramics, clays, alternately carbon nanotubes. Polymer matrix nanocomposites incorporate thermoplastic/thermoset polymer/layered silicates, polyester/TiO₂, polymer/CNT, polymer/layered twofold hydroxides. The majority regularly utilized nano-particles include: Montmorillonite organoclays (MMT), carbon nanofibers (CNFs), Polyhedral Oligomeric Silsesquioxane (POSS), carbon Nanotubes multiwall (MWCNTs), Small-diameter (SDNTs), Also Single-Wall (SWCNTs), Nanosilica (N-silica), Nano aluminum oxide (Al₂O₃), Nano titanium oxide (TiO₂) also how. Thermo-sets and thermo-plastics utilized as matrices for settling on nanocomposites include: Nylons, Polyolefin, e.g. Polypropylene, Polystyrene, ethylene-vinyl acetic acid derivation (EVA) copolymer, epoxy resins, Polyurethanes, Polyamides, Poly Ethylene terephthalate (PET).

C. Synthesis of Al - Nano Al₂O₃ Composites

In general, there are two processing routes to incorporate nano particulates into aluminium. Introduction of particulates into the matrices via ingot casting and powder metallurgy (PM) processes are most popular [7]. Another classification of processing aluminium matrix composites is ex-situ and in-situ methods of fabrication. In 'ex-situ' MMCs the ceramic particulates are synthesized separately prior to the composite fabrication, which often results in agglomeration of fine ceramic particulates during processing. The in-situ route provides several advantages over conventional ex situ process, e.g. in situ formed reinforcements are more uniformly distributed, finer in size, and thermodynamically stable leading to superior mechanical properties compared to their ex situ counterparts [8]. The powder metallurgy route in case of oxide reinforcements has an added advantage as they follow the energy efficient method [9].

1) *Properties of Al/Al₂O₃ Nanocomposites:* Due to their 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 [10]. If compared with pure Al, 2.0 vol% nano-Al₂O₃ additions improves yield strength of around 66%, hardness of around 50% and tensile strength of around 80% [9]. In another study, ultrasonic assisted casting method was used to disperse 2.0 wt% nano-Al₂O₃ (10 nm) in aluminium matrix. Compared with pure Al cast by the same method, composite hardness was increased by 92% and the yield strength by 56% [11]. Owing to low density, low melting point, high specific strength and thermal conductivity of aluminium, a wide variety of reinforcement particulates such as Al₂O₃, SiC, B₄C, AlN, Si₃N₄, TiC, TiO₂, TiB₂ and graphite have been reinforced into it. Among these particulates, Al₂O₃, SiC, B₄C, TiB₂ additions improved the wear behaviour of aluminium matrix composites [12].

II. PROBLEM FORMULATION

A. Proposed work

The problem is associated with the development of Al-Nano Al₂O₃ Composite. 1, 2 & 3 wt % of Nano Al₂O₃ which is fabricated by powder metallurgy technique.

For achievement of the above, an experiment set up was prepared to facilitate the fabrication of required Nanocomposites. The microstructural analysis has been done by optical microscope and SEM to verify the dispersion of reinforcement in the matrix.

B. Objectives

- 1) SEM Analysis of Al Powder & TEM Analysis of Nano Al₂O₃ particulates.
- 2) Fabrication of Al-Nano Al₂O₃ Composite with 1, 2 & 3 weight % of Nano Al₂O₃ particulates.
- 3) Microstructural Analysis of Al-Nano Al₂O₃ Composite with 1, 2 & 3 weight % of Nano Al₂O₃ particulates.

III. EXPERIMENTAL WORK:

A. Cold isostatic compaction chamber

Cold Isostatic Compaction Chamber is used for isostatic compaction of metal, alloys and composite powders at room

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temperature. It consists of 3 pieces: Ejector, Die (Oil Chamber) & Punch

1) Design of Cold Isostatic Compaction Chamber

Given that

$$P_i = 600 \text{ MPa}$$

$$S_{ys} = 684 \text{ MPa (yield strength of EN 10269)} \quad D_i = 50 \text{ mm}$$

$$D_j = 110 \text{ mm}$$

$$\text{F.O.S} = 2$$

In the case of single thick cylinder, if the internal pressure acting on the shell is equal to or greater than the allowable working stress for the material of the shell, then no thickness of the shell will prevent failure. Thus it is impossible to design a single cylinder to withstand internal pressure equal to or greater than the allowable working stress. Therefore we have used compound cylinder design.

In Compound cylinder design, the E.N. 10269 material is used for fabrication of the chamber. It is a ductile material. So we use Maximum Shear Stress Theory. This theory predicts that the yield strength in shear is half of yield strength in tension i.e.

$$S_{ys} = .5 S_{yt}$$

$$\begin{aligned} \text{Allowable stress} &= 2S_{ys} / \text{F.O.S} \\ &= 2 \times 684 / 2 \\ &= 684 \text{ MPa} \end{aligned}$$

We also know that

$$\begin{aligned} \text{Allowable stress} &= P_i \times D_i^2 \frac{\frac{D_o^2}{D_i^2} + 1}{D_o^2 - D_i^2} \\ 684 &= 600 \times 50^2 \frac{\frac{D_o^2}{50^2} + 1}{D_o^2 - 50^2} \\ D_o &= 195.48 \text{ mm} \\ D_o &= 196 \text{ mm} \end{aligned}$$

B. Fabrication of cold isostatic compaction chamber

- 1) *Step 1 Selection of Material / Material Procurement of Required Size:* There are many steel series available in market but EN series is best for manufacturing of Cold Isostatic Compaction chamber. In EN series, there are many codes like EN 24, EN 36, EN 10269 etc. EN 10269 is the better than EN 24 & EN 36. Therefore EN 10269 is used for fabrication of cold isostatic compaction Chamber. Table I shows the properties of EN 10269.

Table I PROPERTIES OF EN 10269

PROPERTIES	$d \leq 100 \text{ mm}$	$100 \geq d \leq 160 \text{ mm}$
Ultimate tensile strength	820-1000 N/mm ²	820 – 1000 N/mm ²
0.2 % proof stress	$\geq 660 \text{ N/mm}^2$	$\geq 660 \text{ N/mm}^2$
Elongation on Gauge Length	$\geq 15 \%$	$\geq 15 \%$
Reduction in Area	$\geq 50 \%$	$\geq 50 \%$

- 2) *Step 2. Roughness Operation:* Roughing Operation was done (allowance of 1.2 to 1.5 mm was provided on all the sides) with the help of power hacksaw .
- 3) *Step 3. Heat Treatment:* This process involves following steps given below,

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- a) *Hardening*: Hardening is a process in which steel is heated to austenite temperature, held at this temperature & quenched in water, oil or molten salt baths. Firstly parts of cold isostatic compaction Chamber were heated up to 880 °C for 3 hours then oil quenching was performed.
- b) *Tempering*: Tempering consist in heating the hardened steel to a temperature below the lower critical temperature, holding it for some time and cooling slowly. After hardening parts were held at 630 °C for 2.5 hours. Then air cooling was performed.
- 4) Step 4 Turning of punch on Lathe machine.
- 5) Step 5 Turning of Outer Cylinder on Lathe machine to the required length & diameter.
- 6) Step 6 Rough Boring of outer & inner cylinder.
- 7) Step 7 Rough turning of the inner cylinder.
- 8) Step 8 Boring of the internal diameter of the inner cylinder.
- 9) Step 9 Press fitting of internal cylinder into outer cylinder on hydraulic press by applying 2000 kg load.
- 10) Step 10 Final operation: (on cylindrical grinding machine)
 - a) Grinding of Compound Cylinder Bore (through internal grinding machine).
 - b) Grinding of punch to suit compound cylinder bore as per given tolerance dimension.
 - c) At last final Inspection.

IV. FABRICATION OF Al – NANO Al_2O_3 COMPOSITES

Standard samples of Al- Nano Al_2O_3 composites with 1, 2 and 3 weight % of Nano Al_2O_3 were fabricated through powder metallurgy route in the following stages:

A. Particle Size analysis of Al and Nano Al_2O_3 powders

Aluminium powder of approximate 50 μm size range and Nano Al_2O_3 particulates of about 40 to 80 nm were taken separately and used in the present work. Figures 1 shows the scanning electron micrograph of the aluminum & Figure 2 expressed the Transmission electron micrograph of Nano Al_2O_3 . The micrographs show that the aluminum powders are of about 50 μm size range and the Nano Al_2O_3 particulates are of about 40 to 80 nm size.

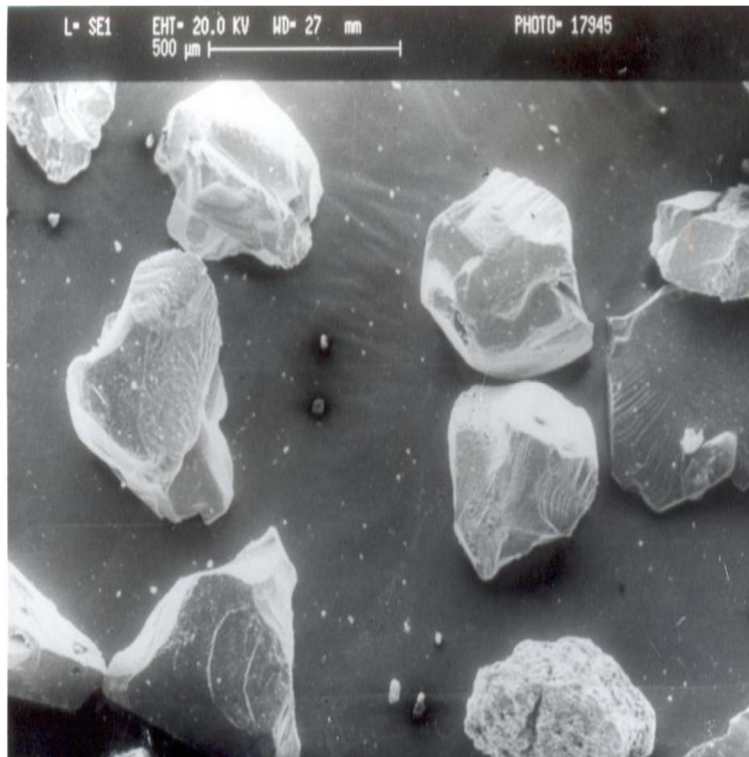


Figure 1 Scanning electron micrograph of the aluminum particles

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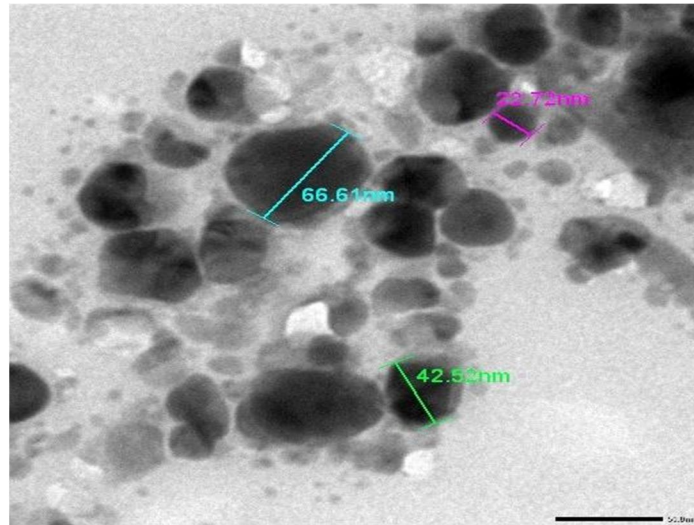


Figure 2 Transmission electron micrograph of Nano Al_2O_3 particles

B. *Mixing of powders*

After particle size analysis, Aluminium & Nano Al_2O_3 powder were weighed individually and mixtures of three different compositions (viz. 1, 2 and 3 weight percent of Al_2O_3) were prepared. The mixtures were put in plastic containers and mixing was done manually for half hours to ensure proper mixing.

C. *Mechanical alloying*

Mechanical alloying results in mixing at molecular level. The Nano Al_2O_3 particles got embedded into Al powder particles and powder particles of Al- Nano Al_2O_3 composite have been obtained. The powder mixtures were transferred to horizontal ball mill. The horizontal ball mill already contained the hardened steel balls as milling media. In order to minimize the extreme tendency of aluminium to get itself welded during milling, 2 wt. % of ethyl acetate was added as process control agent. The milling was performed in Argon atmosphere to prevent contamination from atmospheric air. The ball mill was run at 100 rpm for 8 hours. The powder became hot during milling therefore it is allowed to cool for 3-4 hours then it is taken out of the ball mill and stored in plastic containers.

D. *Mould Cavity*

For the manual compaction of Al- Nano Al_2O_3 composites in the shape of solid cylindrical pins of 15 mm diameter and 25 mm length a die and punch of mild steel have been fabricated. The die was lubricated to ensure easy extraction of the powder compacts. The Al- Nano Al_2O_3 composite powders of weighed amount were mixed with 2-wt. % ethyl acetate & Gum and poured into the die. The powder compact was ejected out of the die and put in a flexible mould and its mouth was tied tightly with the help of a string. The flexible mould should be of appropriate size in order to avoid wrinkles on the mould surface. The flexible mould should be 100 percent leak proof to prevent the leakage of oil into the powder sample during isostatic compaction. Al- Nano Al_2O_3 composite specimens were fabricated with ball milled powder samples with 1, 2 & 3 weight % of Nano Al_2O_3 particulates.

E. *Cold isostatic compaction of al – nano al_2o_3 composites*

The Al- Nano Al_2O_3 composites specimens were fabricated using cold isostatic compaction process. This was performed with the help of Universal Testing Machine. Process of Cold Isostatic Compaction Chamber is as follows:

- 1) Chamber is filled with Hydraulic Oil (2T Oil).
- 2) Powder is to be compacted is filled in a flexible mould and Cylindrical shape as per the design is given using mild steel die.
- 3) The flexible mould is tied with string and dipped in Hydraulic oil in the chamber.
- 4) Pressure of 600 MPa is applied with the help of punch on UTM of 100 Ton capacity.

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- 5) After compaction flexible mould is taken out.
- 6) Air is removed & it is once tied again with the string & re-compaction is done.

F. Sintering of *al – nano Al₂O₃ compacts*

The cold isostatically pressed green compacts were sintered in a programmable Argon atmosphere furnace by gradually raising the temperature to 645°C and the specimens were kept at this temperature for 1 Hour and 25 Minutes. The compacts were furnace cooled. Sintering was performed in continuous flow of argon gas.

G. Machining of *sintered al-nano Al₂O₃ compacts*

After the sintering operation, the shape of sintered Al-Nano Al₂O₃ Compacts were irregular from both sides. Sintered Al-Nano Al₂O₃ Compacts were machined to cylindrical shape on the lathe machine.

V. MICROSTRUCTURAL ANALYSIS

The microstructures of the Al- Nano Al₂O₃ composites were studied using scanning electron microscope. For this purpose small samples were cut from the cylindrical pins fabricated by powder metallurgy process. The samples were first machined on Lathe and then polished using polishing papers of gradually increasing fineness. The polished samples were then lapped on polishing machine using diamond-lapping paste and velvet cloth for about 40 minutes so that mirror finish is obtained on the samples. The samples were etched with 1 % Keller Reagent for about 45 seconds and washed with distilled water before the microstructural analysis. Then the scanning electron micrographs of powder metal Al- Nano Al₂O₃ composite samples with 1 to 3 weight % of Nano Al₂O₃ were taken and studied for microstructural analysis.

A. Optical Microscopic Analysis of *Al – Nano Al₂O₃ Composites*

Microstructure was visualized with the help of optical microscope. For specimen preparation first of all specimen were cut down into small cylindrical shapes after that different samples were grinded on different grit size emery papers sequentially by 100, 220, 660, 1000. After grinding specimen were polished on polishing machine by alumina paste and then etched by kellar's reagents to better contrast. The specimen was visualized on different magnification 200X to show presence of reinforcement and its distribution on the metal matrix composites. The microstructures of all samples are shown in figure.

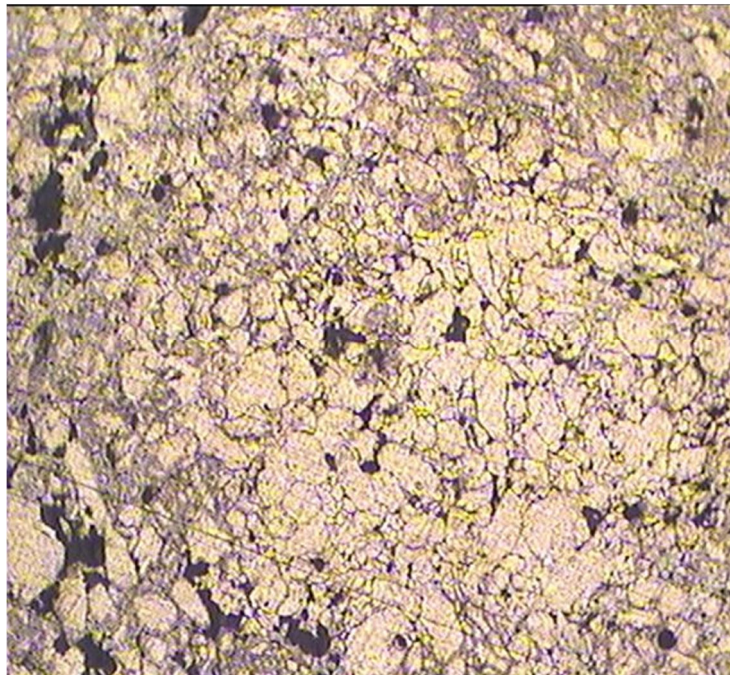


Figure 3 Optical Micrographs of Al-1 wt. % Nano Al₂O₃ Composite after sintering

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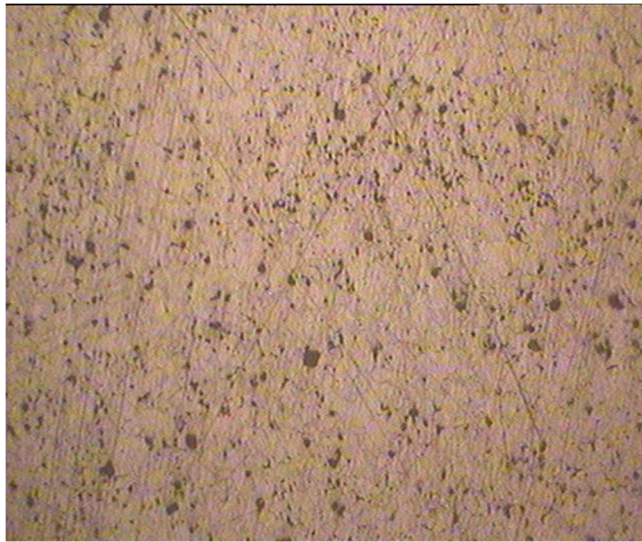


Figure 4 Optical Micrographs of Al - 2 wt. % Nano Al_2O_3 Composite after sintering

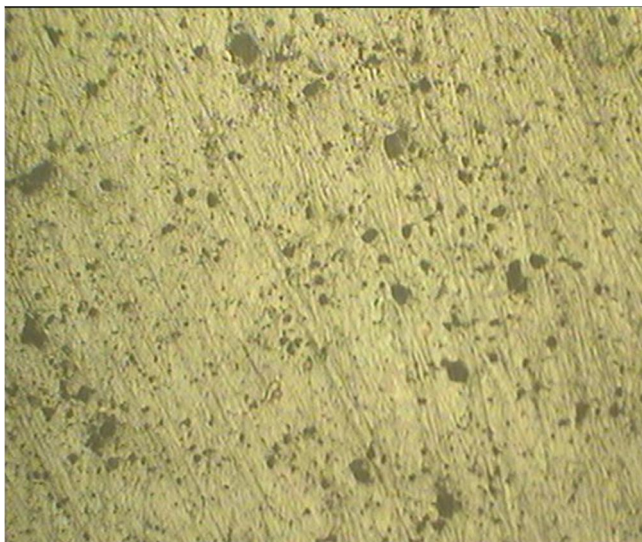


Figure 5 Optical Micrographs of Al - 3 wt. % Nano Al_2O_3 Composite after sintering

It is clear from figure 3 to 5 distribution of alumina particulates in a matrix alloy fairly uniform. The shape of Al_2O_3 particles are irregular in nature. Irregular shape of Al_2O_3 particles may be due to breakages of particles during the ball milling.

It is found that as the percentages of reinforcement increases the area fraction also increases as shown in the optical micrographs. It is also observed that there is increase in hardness and mechanical properties, this can be attributed to the increase in interfacial bonding of reinforcement with the aluminium matrix. It is also found that at some places there is clustering of Al_2O_3 particulates as shown in figure 5.

1) SEM Analysis of Un-Sintered Al-1, 2 & 3 wt. % Nano Al_2O_3 Composites: Figure 6 shows SEM image of Al-1 wt. % Nano Al_2O_3 Composite. This image shows that the Al-Nano Al_2O_3 are not properly bonded to each other but some moisture is present. Figure 7 shows SEM image of Al-2 wt. % Nano Al_2O_3 Composite. This image shows that the Al-Nano Al_2O_3 are not properly bonded to each other. The SEM images show that nano particles are uniformly distributed in the aluminum matrix. Figure 8 shows SEM image of Al-3 wt. % Nano Al_2O_3 Composite. This image shows that there is clustering of nano particles at some of the places.

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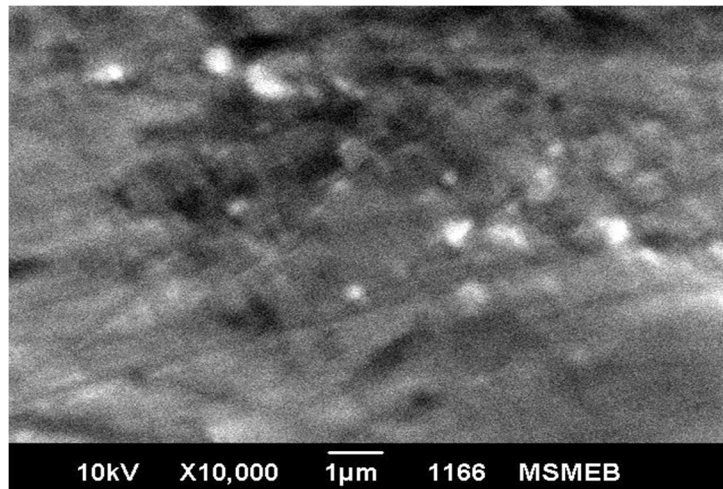


Figure 6 SEM Image of Al-1 wt. % Nano Al₂O₃ Composite before sintering

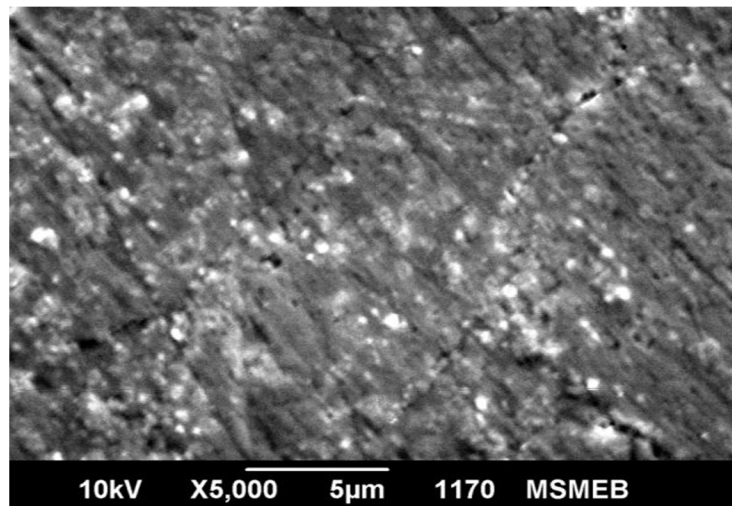


Figure 7 SEM Image of Al-2 wt. % Nano Al₂O₃ Composite before sintering

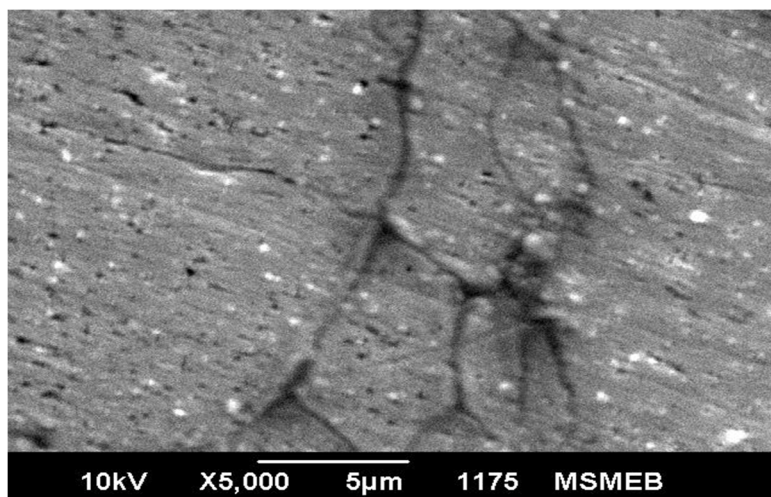


Figure 8 SEM Image of Al-3 wt. % Nano Al₂O₃ Composite before sintering

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B. SEM Analysis of Sintered Al-1, 2 & 3 wt. % Nano Al_2O_3 Composites

The etched and dried specimens were prepared and subsequently mounted on specially designed aluminium stubs using holder. The specimens thus mounted were viewed under Jeol JSM 6510 LV scanning electron microscope at a voltage of 10 kV. Figure 9-11 show the SEM micrograph of the different Al- Nano Al_2O_3 composites.

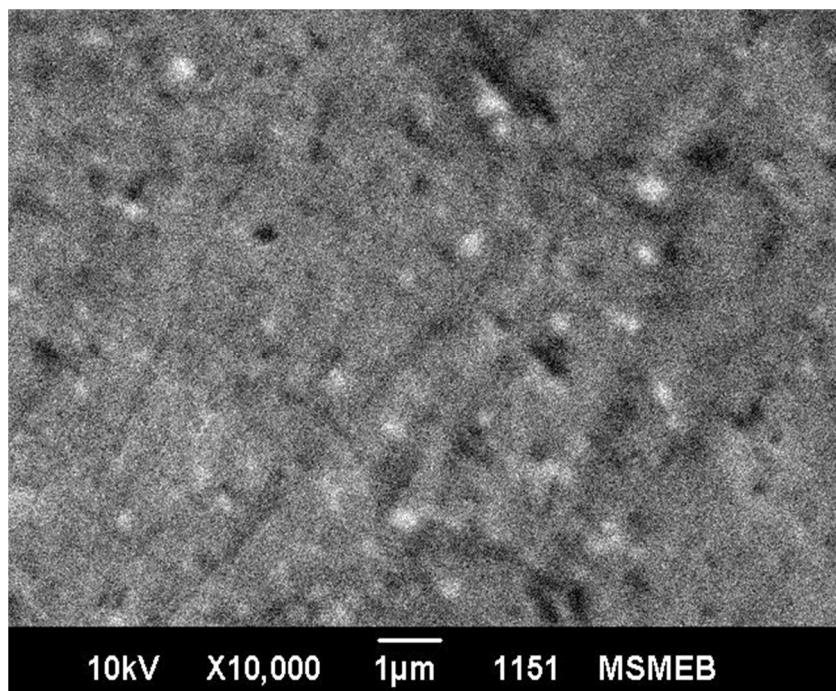


Figure 9 SEM Image of Al-1 wt. % Nano Al_2O_3 Composite after sintering

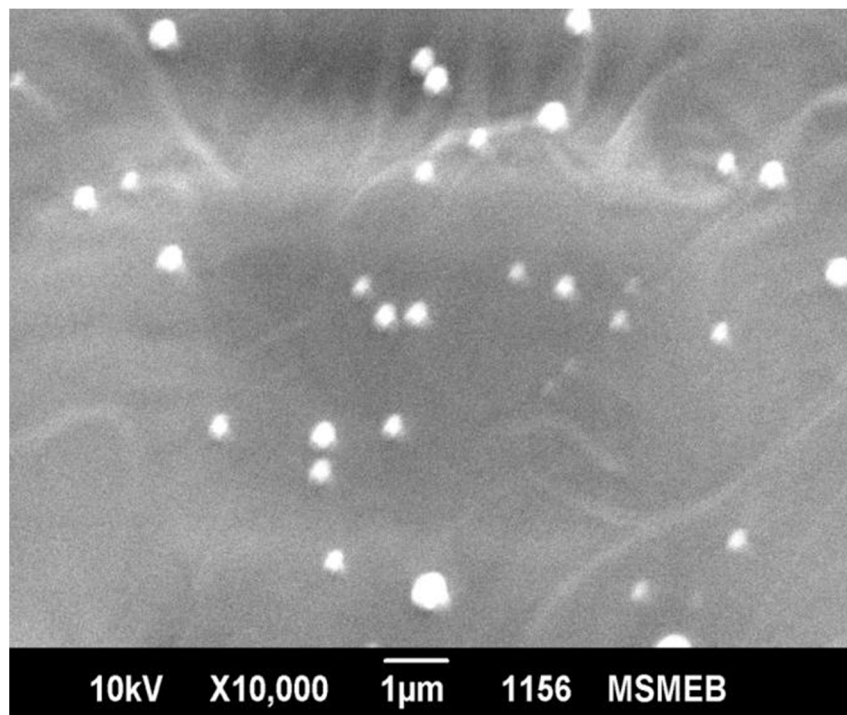


Figure 10 SEM Image of Al-2 wt. % Nano Al_2O_3 Composite after sintering

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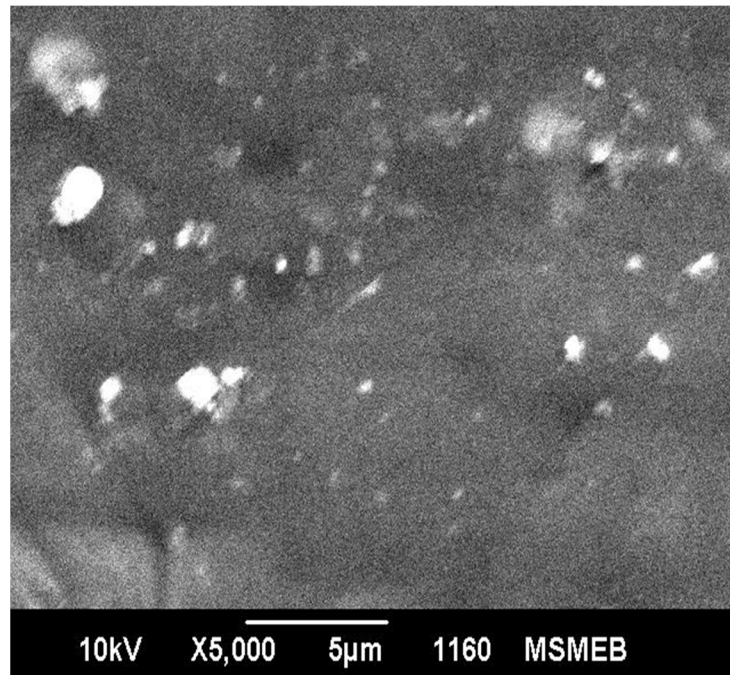


Figure 11 SEM Image of Al-3 wt. % Nano Al_2O_3 Composite after sintering

Figure 9 shows SEM image of Al-1 wt. % Nano Al_2O_3 Composite. This image shows that the Al-Nano Al_2O_3 are properly bonded to each other. Figure 10 shows SEM image of Al-2 wt. % Nano Al_2O_3 Composite. This image shows that the Al-Nano Al_2O_3 are properly bonded to each other but some extent greater than former one. The SEM images show that nano particles are uniformly distributed in the aluminum matrix. Figure 11 shows SEM image of Al-3 wt. % Nano Al_2O_3 Composite. This image shows that there is clustering of nano particles at some of the places.

VI. CONCLUSION

A. Cold isostatic compaction at 600 MPa followed by Argon atmosphere sintering at 645°C has been successfully used to produce Al-Nano Al_2O_3 composites.

B. Scanning electron micrographs of powder metal Al-Nano Al_2O_3 composites reveals that the Argon atmosphere sintering results in proper bonding between aluminium and Nano Al_2O_3 particles. The micrographs also show some amount of porosity and uniform distribution of Nano Al_2O_3 particulates in aluminium matrix up to 2 wt. % Nano Al_2O_3 .

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