# Development and characterizations of aluminum metal matrix hybrid composite

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By

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*Dedicated*
*to my beloved family*

# Certificate

This is to certify that the thesis entitled **"Development and characterizations of aluminum metal matrix hybrid composite**" has been carried out by **Mr.Souvik Bose (Examination Roll No: M4MAT24003 and Registration No. 163731 of 2022-23)** under my guidance and supervision and accepted in partial fulfillment for the degree of Master of Technology in Metallurgical Engineering from Jadavpur University. To the best of our knowledge the content of this thesis or any parts thereof have not been previously submitted for the award of any degree or diploma.

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# Declaration of Originality and Compliance of Academic Ethics

I hereby declare that this thesis “**Development and characterizations of aluminum metal matrix hybrid composite** ” contains literature survey and original research work by me, as a part of my M.TECH. Degree in Metallurgical Engineering during the academic session 2022-2024. All information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by this rules and conduct, I have fully cited and referred all material and results that are not original to this work.

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The foregoing thesis is hereby approved as a creditable study of an engineering subject and presented in a manner satisfactory to warrant acceptance as a pre- requisite to the degree for which it has been submitted. It is understood that by this approval the undersigned does not necessarily endorse or approve any statement made, opinion expressed or conclusion drawn there in but approve the thesis only for which it is submitted.

Committee on final examination for the evaluation of the thesis

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Signature of Examiners

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# Abstract

*This thesis delves into a comparative exploration of aluminum matrix hybridcomposites, bolstered by nickel nanoparticles, focusing on their microstructural nuances and mechanical attributes. Employing magnetic nickel (Ni) nanoparticles, synthesized via the sol-gel technique with an average size of 31.20 nm, as the reinforcing agents, the study investigates their integration into commercially pure aluminum matrix. Utilizing the powder metallurgy process, aluminum matrix samples with varying nickel weight percentages (2, 5, 7, and 10 wt% of aluminum) of ceramic reinforcement in relation to aluminum were crafted. Sintering at 650°C was conducted to refine the density and uniformity of the composite structure. Assessment techniques encompassed optical microscopy, SEM, and XRD analyses, facilitating the evaluation of microstructure, porosity distribution, density and potential matrix-reinforcement interactions. Findings indicated a decline in the relative density of the composites with increasing reinforcement weight percentage, alongside a moderate enhancement in hardness.*

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# CHAPTER-1

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# *INTRODUCTION*

Aluminum is one of the important plentiful metal on Earth's crust. It is attractive for its low density, light weight, ease of machining, resistance to corrosion and availability. Because of its numerous desirable qualities, aluminum is the most widely used, readily and has been for many hundreds of thousands of years in academics and industry, particularly in electrical, electronics, automotive, aerospace and so on.

The low melting point and lack of strength restrict the use of pure aluminum. In order to improve the strength and stiffness of pure aluminum, promise has been found for shorting out these issues by adding hard metal and/or ceramic reinforcements. Addition of magnetic material with ceramic reinforcement can lead aluminium for versatile applications.

One significant class of magnetic materials are ferromagnetic material. Regarding the ongoing advancement of magnetic materials, a number of ferromagnet related research projects over the past few decades have produced creative outcomes including theories, models, and cutting-edge preparation techniques. Ferromagnet are commonly used in transformers, motors, generators, sensors and telecommunication engineering. Ferromagnets are typically outperform other materials because of their unique qualities, which include high magnetic capabilities, low cost, and practical applicability. Iron, cobalt and nickel are the members of ferromagnetic materials.

Conventional magnetic materials often suffer from significant energy losses due to hysteresis and eddy currents, reducing their efficiency. Their performance can degrade at high temperatures, limiting their applications in extreme environments. They are generally bulky and heavy, posing challenges in miniaturizing devices. Manufacturing these materials can be expensive and resource-intensive. Additionally, they can be susceptible to demagnetization over time or when exposed to external magnetic fields.

Composite magnetic materials offer superior performance with reduced energy losses due to lower hysteresis and eddy currents. They maintain stability and efficiency at high temperatures, expanding their usability in harsh environments. These materials are lighter and can be tailored to specific applications, facilitating miniaturization of devices. Their production can be more cost-effective and resource-efficient. Additionally, they exhibit improved resistance to demagnetization and external magnetic fields.

Present research efforts to develop and characterize the aluminum silica nickel magnetic hybrid composite. Various compositions of aluminum silica nickel based hybrid composites have developed through Powder Metallurgy technique. Finally, optimised the sample based on its various physical and mechanical properties.

# CHAPTER 2

# *LITERATURE REVIEW*

## 2.1 Composite

### 2.1.1 Why composite?

A typical composite material comprises two or more materials mixed and bonded on a macroscopic scale. For instance, concrete combines cement, sand, stones, and water. On a microscopic level, if the composition occurs at the molecular level, the resulting material is termed an alloy for metals or a polymer for plastics. Generally, composite materials consist of reinforcement, such as fibers, particles, flakes, or fillers, embedded in a matrix of polymers, metals, or ceramics. The matrix serves to hold the reinforcement in place to achieve the desired shape, while the reinforcement enhances the overall mechanical properties of the matrix. Properly designed, this combined material exhibits superior strength compared to each individual component

### 2.1.2 Matrix phase and Dispersed phase

**Matrix phase-**The matrix phase in composites is the continuous body constituent that tends to enclose the composite and gives the bulk form to the composite material. This phase can be a polymer, metal, or ceramic material. Basically, the matrix is a homogenous and monolithic material in which we can observe a fiber system of a composite that is embedded. This phase is completely continuous, and it provides a medium for binding and holding reinforcements together to form the solid material.

- Composite reinforced by particle
- Composite reinforced by chopped strands
- Unidirectional composites
- Laminates
- Fabric reinforced plastics
- Honey comb composite structure

**Dispersed phase-**The dispersed phase in composites is the discontinuous phase of the composite material that determines the internal structure. We can call this phase the phase that is dispersed in colloidal particle shape. Moreover, the medium in which the colloidal particles are distributed is known as the medium of dispersion. Typically, the dispersed phase is also known as the reinforcing phase, and it is held together by the matrix phase. The dispersed phase improves the overall properties of the matrix. It is strong if the reinforcement provides a low density

The matrix phase in composites is the continuous body constituent that tends to enclose the composite and give the bulk form to the composite material. The dispersed phase in composites is the discontinuous phase of the composite material, which determines the internal structure. Therefore, the key difference between matrix and dispersed phase in composites is that the matrix phase is a continuous phase, whereas the dispersed phase is a discontinuous phase in composites.

### 2.1.3 Advantages of composite

Lightweight: Composites offer a notable advantage in weight compared to most woods and metals. This characteristic is particularly crucial in industries such as automotive and aerospace, where reduced weight translates to improved fuel efficiency, allowing for more miles per gallon. Aircraft designers place significant emphasis on weight reduction, as lighter crafts require less fuel and can achieve higher speeds. Some modern aircraft, like the Boeing 787 Dreamliner, incorporate more composites than metal.

High Strength: Composites can be engineered to possess far greater strength than aluminum or steel. Unlike metals, which exhibit uniform strength in all directions, composites can be tailored to be strong in specific orientations.

Strength-to-Weight Ratio: This ratio signifies a material's strength relative to its weight. While some materials, like steel, are both strong and heavy, others, like bamboo poles, are strong yet lightweight. Composite materials can be designed to offer both strength and lightness simultaneously, making them ideal for applications like aircraft construction, where exceptional strength is required at minimal weight. Unlike metal, where increased strength often necessitates added thickness and weight, composites can achieve strength without bulkiness.

Corrosion Resistance: Composites demonstrate resilience against weathering and harsh chemicals, which can corrode other materials. They are particularly suitable for environments where exposure to chemicals is common, as well as outdoor applications where they endure severe weather conditions and temperature fluctuations.

High-Impact Strength: Composites can be formulated to absorb impacts effectively, whether from sudden forces like bullets or explosions. Because of this property composites are used in bullet proof vests and panel, and to shield airplanes, buildings etc.

Design Flexibility: Composites offer unparalleled moldability, allowing for the creation of intricate shapes with ease, a capability not easily achieved with other materials. This versatility empowers designers to realize almost any desired form or configuration. For instance, fiberglass composites are widely employed in constructing recreational boats due to their ability to be molded into complex shapes, enhancing both design aesthetics and cost-effectiveness. Moreover, the surface of composites can be molded to replicate a wide range of finishes or textures, from smooth to textured.

Part Consolidation: A single composite component can replace an entire assembly of metal parts, streamlining manufacturing processes and reducing maintenance requirements throughout the item's lifespan. Consolidating parts saves time and lowers maintenance overheads.

Dimensional Stability: Composites maintain their shape and size under varying environmental conditions, unlike materials like wood that expand and contract with changes in humidity. This dimensional stability makes composites preferable for applications requiring consistent fits, such as aircraft wings, where maintaining precise dimensions regardless of altitude fluctuations is crucial.

Nonconductive: Composites do not conduct electricity, making them suitable for applications like electrical utility poles and electronic circuit boards. However, if electrical conductivity is necessary, composites can be engineered to be conductive.

Nonmagnetic: Since composites contain no metals, they are nonmagnetic, making them compatible with sensitive electronic equipment. Their lack of magnetic interference enables the use of large magnets in MRI equipment for better performance. Composite materials are employed in various components of MRI machines and even in the construction of the room, using composite rebar to reinforce concrete walls and floors in hospitals.

Radar Transparent: Composites allow radar signals to pass through unhindered, making them invaluable in radar-equipped environments, whether on the ground or in the air. This property is particularly crucial in stealth aircraft, such as the U.S. Air Force's B-2 stealth bomber, which relies on composites to minimize radar detection.

Low Thermal Conductivity: Composites exhibit excellent insulating properties, effectively mitigating heat or cold transfer. They find application in building components like doors, panels, and windows where enhanced protection against severe weather conditions is required.

Durable: Structures constructed from composites boast a prolonged lifespan and require minimal maintenance, ensuring long-term reliability and cost-effectiveness.

### 2.1.4 Applications composites

Appliance Cooking, Dishwasher, Refrigerator, Small Appliances, Laundry, Ice Machines Construction Entry Doors, Garage Doors, Architecture, Countertops, Wastewater Treatment Electrical Distribution Circuit Breakers, Motor Control, Centers, Generators, Switchgear, Bus way, Control Cabinets, Cross Arms Energy Wind Turbine, Fuel Cells, Solar Panels, Pumps Forward Lighting Headlamps, Reflections HVAC Drain Pans, Blower Housing, Wall Sleeves, Control Panels, Recreational Vehicles Lighting Class 1/ DV2, Light Housing, In ground, Explosion Proof, Reflectors Marine Engine Covers, Personal Watercraft, Boat Access Covers, Electrical Buyer's, Motor Housing Sanitary/Plumbing Faucets, Sinks, Drains, Showers, Bathtubs Transportation Resisters Drive Motors, Controls, Valve Covers, Oil Pans, Air Suspensions and in many more places.

In summary, composites possess numerous desirable properties, rendering them highly versatile and ideal for a wide array of applications. These exceptional qualities often surpass those of traditional materials currently in use. Consequently, there is a pressing need for extensive research and advancement in the field of composite materials to harness their full potential.

## 2.2 Hybrid composite

The utilization of fiber-reinforced plastics is experiencing a steady rise, accompanied by an expanding array of available fiber and resin systems for designers. While certain systems excel in specialized applications, prioritizing qualities like low density, high rigidity, and strength, they may also present limitations such as high cost and susceptibility to brittle fracture. By blending multiple fiber types within a resin to form hybrid composites, it becomes feasible to harness the combined advantages of individual components while mitigating their less desirable attributes. Additionally, tailoring the properties of these materials to specific requirements is achievable. Many scenarios demand high-modulus materials, yet traditional brittle failure is unacceptable. For instance, in structural elements like strut members, a high initial modulus followed by controlled yielding and minimal reduction in load-carrying capacity is preferred. Hybrid fiber-reinforced materials can be produced through two methods: intimately mingling fibers in a common matrix or laminating alternate layers of each composite type.

Metal matrix composites (MMCs) reinforced with ceramic particles exhibit significant promise for structural applications owing to their excellent blend of properties. These composites merge the desirable traits of metallic alloys, such as ductility and toughness, with the advantageous characteristics of ceramic reinforcements, including high strength and modulus. This combination results in a superior profile of characteristics.

Among MMCs, aluminum matrix composites (AMCs) stand out, possessing properties like low density, high stiffness and strength, superior wear resistance, controlled coefficient of thermal expansion, enhanced fatigue resistance, and improved stability at elevated temperatures. Consequently, they find utility in a wide array of advanced applications. Notably, the utilization of AMCs in engine applications has shown potential to reduce overall weight, fuel consumption, and pollution in automobiles and aircraft.

AMCs reinforced with either silicon carbide (density: 3.18 g/cm$^3$) or alumina (density: 3.9

g/cm³) particles emerge as attractive materials for such applications. Although these reinforcements are denser than those of aluminum alloys (density: 2.7 g/cm³), they enhance the weight of the composites depending on the reinforcement content. Additionally, incorporating ceramic particles into the Al-alloy boosts the composite's hardness but complicates machining processes. Such challenges can be addressed by employing multiple reinforcements in the aluminum alloy. Ceramic reinforcements offer superior strength compared to other types of reinforcements, making them the primary choice for enhancing the properties of these composites.

However, the incorporation of secondary reinforcements offers cost reduction and weight reduction benefits due to their ready availability and lower density in hybrid composites. The properties of both primary and secondary reinforcements can be combined to optimize material properties. Additionally, the use of stir casting technique for AMC fabrication contributes to cost reduction as it is an economical, simple, and highly productive method.

Currently, there is a growing interest in producing Hybrid Aluminum Matrix Composites (HAMCs) with enhanced physical and mechanical properties. Recent research suggests that agro/industrial waste materials like fly ash, graphite, rice husk ash, etc., can serve as complementary reinforcements in AMCs. Moreover, the incorporation of two ceramic phases can further enhance hybrid composites' development.

Employing hybrid reinforcements improves composite performance by introducing new features. Firstly, it can lower the cost of aluminum composites. Secondly, it enables control over composite weight due to the lower density of these materials. Thirdly, these composites can offer comparable or even superior physical and mechanical properties.

### 2.2.1 Application of aluminum hybrid composite and future scope

Presently, material researchers are giving significant attention to designing high-performance, low-cost components. This objective can be achieved by incorporating industrial and agro-waste materials as green reinforcements in aluminum matrix composites. The disposal of these materials poses environmental challenges, thus recycling them to create green materials has become a focal point of current research efforts. Due to their environmental friendliness, energy efficiency, and cost-effectiveness, these materials show promising market potential as reinforcement materials for composites.

Various waste materials such as fly ash, red mud, palm oil fuel ash (POFA), palm oil clinker (POC), rice husks, coconut husks, and sugarcane bagasse have the potential for utilization in construction and automotive industries. Extensive research has been conducted, and ongoing developments aim to successfully utilize waste materials as partial reinforcements in composites.

The present study demonstrates that Hybrid Aluminum Matrix Composites (HAMCs) offer a unique combination of mechanical and physical properties that are rarely achievable with ceramic-reinforced composites. HAMCs can find applications in automobile, aircraft, marine, defense, sports, recreation, and various other industries. Notable advantages of HAMCs include their relatively low cost, lightweight, and higher strength-to-weight ratio compared to ceramic-reinforced composites.

However, investigations into the wear properties of these composites need to be conducted under different parametric conditions, as there is limited literature available on this aspect. In conclusion, there is immense potential, scope, and opportunities for researchers in predicting and improving the characteristics of HAMCs.

### 2.2.2 Challenges opportunity and future trends in hybrid composite

To enhance the engineering utilization of hybrid composites, several challenges need to be addressed. Overcoming these challenges requires concerted efforts in design, research, product development, and business development. The following issues must be prioritized:

1. Understanding the science of primary processing of hybrids comprehensively, especially factors influencing microstructural integrity.
2. Improving damage-tolerant properties, particularly fracture toughness and ductility, in hybrid composites.
3. Exploring methods to produce high-quality, low-cost reinforcements from industrial wastes and byproducts.
4. Focusing efforts on developing hybrids based on non-standard fibers and matrices.

5. Establishing a classification system for different grades of hybrids based on property profiles and manufacturing costs.
6. Developing simple, economical, and portable non-destructive kits to quantify undesirable defects in hybrid composites.

In conclusion, the applications of hybrid composites span various industries, including transportation, aeronautics, naval, automotive, and electronics. Extensive research efforts have been directed towards understanding the manufacturing processes of hybrid laminates and their application in cutting-edge technologies. Glass and carbon fiber-reinforced composites have received equal attention, with epoxy resin being the preferred matrix material. This literature on hybrid composites serves as a valuable resource for researchers and scientists involved in advancing hybrid composite technology.

## 2.3 Nanocomposite

The realm of technology relies heavily on material science, spanning across energy devices, automobiles, sensors, medicine, and structural components. Traditional materials like metals, ceramics, and polymers come with inherent limitations. To overcome these, a composite approach merges two or more materials to enhance functionalities, often reducing one component to the nanoscale (1–100nm) for superior properties. Nanocomposites, with their increased surface area and quantum effects, exhibit enhanced electrical, mechanical, chemical, optical, and magnetic characteristics, making them pivotal in modern research and everyday applications.

Comprising a matrix infused with tailored fillers, nanocomposites utilize both organic and inorganic materials. They can be categorized into polymer matrix nanocomposites, ceramic matrix nanocomposites, or metal matrix nanocomposites based on their matrices. These materials, synthesized from multiphase solids, blend components within nanoscale dimensions, featuring continuous and discontinuous phases.

Given the significance of nanocomposite materials, numerous studies focus on refining synthesis techniques. Among the types of nanocomposites, ceramic matrix nanocomposites like silica nickel variants are prominent. Synthesis methods for these materials vary, with the aim to enhance their properties and applications.

Our research centers on synthesizing silica nickel nanocomposites through sol-gel techniques, mix it with aluminum powder in different weight percentage in powder metallurgy techniques and characterizing them comprehensively.

### 2.3.1 Different types of nanocomposite

There is actually three types of nanocomposite firstly polymer nanocomposite,secondly ceramic nanocomposite and thirdly metal matrix nanocomposites.

#### Polymer nanocomposite

Polymers have found widespread use across various industries due to their ease of processing, lightweight nature, and ductility. Nonetheless, they do possess certain drawbacks, including low strength, inferior mechanical properties, and reduced stability compared to ceramics and metals. Polymer nanocomposites, featuring nanoparticle or nanofiber-based fillers, offer a solution to these limitations and are emerging as highly efficient materials for applications in aerospace components, sports gear, solar cells, and supercapacitors . These nanocomposites maintain the advantageous characteristics of polymers such as low weight, processability, and ductility, while simultaneously enhancing mechanical strength, gas impermeability, heat resistance, biodegradability, and other properties through the incorporation of fillers.

#### Ceramic nanocomposite

A ceramic matrix nanocomposite primarily consists of ceramics, chemical compounds formed from oxides, nitrides, silicides, and similar materials, with metal serving as the secondary component. Typically, ceramic matrix nanocomposites are fabricated using various techniques. Among these, "Powder Process" methods are extensively employed for the synthesis of ceramic nanocomposites**.**

#### Metal matrix nanocomposite

Nanomaterials featuring a resilient metallic matrix with either pliant reinforcement or sturdy ceramics are referred to as metal-matrix nanocomposites. These materials present numerous advantages compared to monolithic metals, such as elevated specific strength and modulus, reduced coefficients of thermal expansion, and enhanced wear resistance.

### 2.3.2 Various techniques in synthesis of ceramic matrix nanocomposites

A ceramic matrix nanocomposite primarily consists of ceramics, chemical compounds formed from oxides, nitrides, silicides, and similar materials, with metal serving as the secondary component. Typically, various techniques are employed for the preparation of ceramic matrix nanocomposites. Among these, "Powder Process" methods are extensively utilized in synthesizing ceramic nanocomposites. Some of the techniques suitable for the preparation of different types of ceramic nanocomposites by powder process are shown in the following figure.

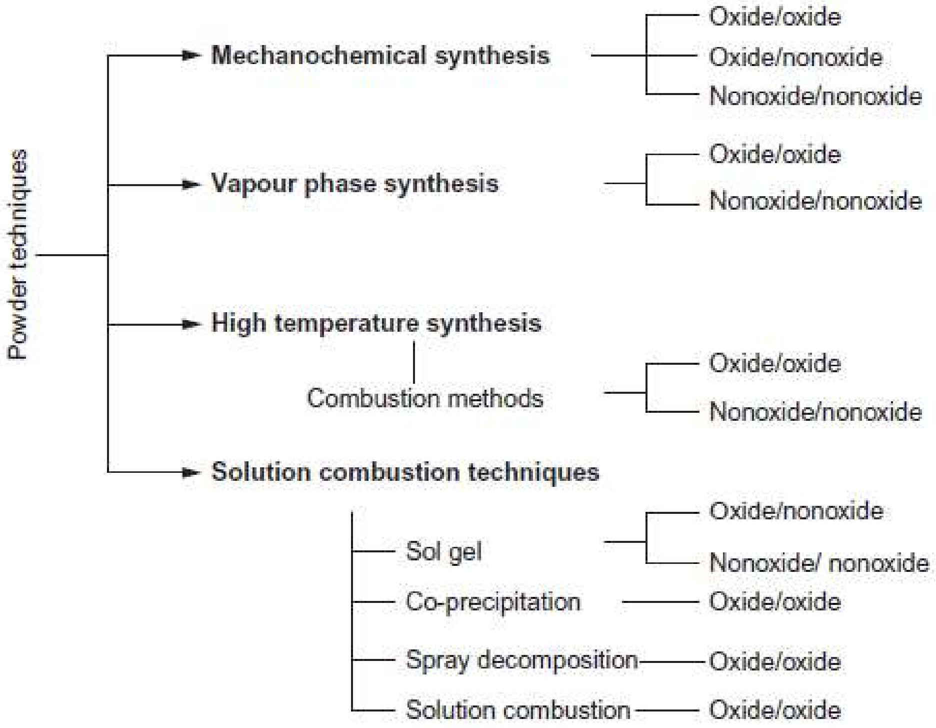

**Figure** 2.1 : Various powder process techniques for the synthesis of ceramic nanocomposites

**Sol-Gel**

Sol-gel is one of the simple wet chemical techniques widely used for the preparation of nanocomposites (mostly oxides) and ceramics. The sol-gel process is a chemical transformation of a sol into gel (three-dimensional polymer) state

and a subsequent transition into solid oxide material caused by suitable post treatment. This method is based on inorganic polymerization reaction including hydrolysis, polycondensation, gelation, aging, drying, and calcination or sintering (densification). The sol-gel synthesis steps for the preparation of oxide nanocomposites are
given below:

**1. Hydrolysis**: When the alkoxide precursor (M-OR) is hydrolyzed by mixing with water or alcohol, metal hydroxide is formed. The total reaction can be expressed as
$M(OR)x + x/2H2O=MOx/2 + xROH$

**2. Poly condensation**: The two metal hydroxides can link together in the condensation reaction by the removal of $H_2O$ and R-OH.
$M\text{-}OR + M\text{-}OH=M\text{-}O\text{-}M +ROH$
The rates of hydrolysis and condensation processes are crucial factors that determine the characteristics of final products. Slower and more controlled hydrolysis normally results in smaller particle size with unique properties.

**3. Gelation**: The gelation time (the time taken for the formation of gel) depends on the following parameters: temperature, solvent, pH condition, and removal of the solvent. At the gel point, a rigid substance, called an alcogel, is formed, which consists of solid and liquid parts. The solid part is formed by the three-dimensional network of linked oxide particles and
the liquid part fills the free space surrounding the solid part.

**4. Aging**: During the aging of the alcogel for a certain period of time, the polycondensation continues, leading to the expulsion of liquid from the pores. Hence, the thickness of particle necks increases and the porosity decreases. Thus, the aging process increases the strength of the gel.

**5. Drying**: During the drying process, the liquid existing in the interconnected pore network is removed. Thus, there is a decrease in the volume of the gel, which is equal to the volume of the liquid lost by evaporation. The resulting monolithic is termed a xerogel (xero-dry). If the drying process is carried out under supercritical conditions, the product is called an aerogel.

**6. Dehydration or chemical stabilization**: In this step, the unwanted elements like H and R are removed from M-OH and M-OR bonds, respectively, to obtain a chemically stable compound.

**7. Densification**: This is the final process of the technique, in which the pores are eliminated and a densified polycrystalline can be obtained by heating the porous gel at high temperatures.

### 2.3.3 Various techniques in synthesis of metal matrix nanocomposite

Metal-matrix nanocomposites are composed of strong ceramics or tough metallic nanoparticles with soft reinforcement. Compared to monolithic metals, metal matrix nanocomposites provide a number of benefits, including as greater wear resistance, lower coefficients of thermal expansion, and higher specific strength and modulus.

Depending on the metallic matrix's processing temperature, the techniques used to create MMCs can be divided into the five groups listed below: There are five types of processes that can be used: (1) liquid-phase processes, (2) solid-phase processes, (3) two-phase processes, (4) deposition techniques, and (5) in situ procedures.

**Liquid-Phase Processes** involve the integration of ceramic particulates into a molten metallic matrix using various proprietary methods. The subsequent stages encompass mixing and eventually casting the resultant composite mixture. Criteria for selecting a ceramic reinforcement include factors such as matrix alloy, melting temperature, tensile strength, thermal stability, elastic modulus, size and shape of reinforcing particles, and cost considerations.

**Solid-Phase Processes** entail the preparation of particulate-reinforced Metal Matrix Composites (MMCs) from blended elemental powders. Powder metallurgy is the primary technique falling within this category. This process involves steps such as powder preparation, blending, compacting, sintering, testing, sizing, and machining.

**Two-Phase Processes** involve mixing ceramic and matrix materials within a region of the phase diagram where the matrix contains both solid and liquid phases. Major techniques within this category include spray deposition and compocasting/rheocasting.

**Deposition Techniques** encompass methods such as immersion plating, electroplating, spray deposition, Chemical Vapor Deposition (CVD), and Physical Vapor Deposition (PVD). In these techniques, individual fibers are coated in conjunction with the matrix material required to form the composite. Subsequent diffusion bonding results in the formation of a consolidated composite plate or structural shape.

**In Situ Processes** involve the formation of the reinforced phase within the matrix material during processing. These techniques allow for the production of the composite material in a single step from an appropriate starting alloy.

## 2.4 Powder properties

### 2.4.1 What is powder?

Powder constitutes a dry, voluminous solid comprising numerous exceedingly minute particles capable of free flow when agitated or inclined. While powders represent a distinct subset of granular materials, the terms "powder" and "granular" are occasionally employed to differentiate between distinct material categories. Specifically, powders denote those granular materials characterized by finer grain sizes, thus exhibiting a heightened propensity for clump formation during flow. On the other hand, granulars encompass the coarser granular materials that typically avoid clumping unless subjected to moisture.

### 2.4.2 Density and Porosity

**Density-** Density, also known as volumetric mass density or specific mass, represents the mass of a substance per unit volume. The commonly used symbol for density is ρ (the Greek letter rho in lowercase), although the Latin letter D can also be utilized. Mathematically, density is expressed as the ratio of mass to volume:

$\rho = m/V$

Where $\rho$ represents density, m stands for mass, and V denotes volume.
Here we use metal powder so generally we use bulk density. The bulk density of a powder is the ratio of the mass of an untapped powder sample and its volume including the contribution of the interparticulate void volume.

**Porosity-**
Porosity or void fraction is a measure of the void (i.e. "empty") spaces in a material, and is a fraction of the volume of voids over the total volume, between 0 and 1, or as a percentage between 0% and 100%. Strictly speaking, some tests measure the "accessible void", the total amount of void space accessible from the surface

Pores represent the most important defect in polycrystalline ceramics. The presence of pores is usually detrimental to the mechanical properties of bulkceramics, since pores provide a pre-existing location from which a crack can grow. The presence of pores is one of the reasons why ceramics show such brittle behavior under tensile loading. Since there is a distribution of pore sizes and the overall level of porosity changes, the mechanical properties of ceramics vary. This variability is measured using Weibull statistics . The presence of pores, on the other hand, may be useful for increasing resistance to thermal shock. In certain applications, such as filters for hot metals or for liquids or gases, the presence of interconnected pores is desirable. Pores in a ceramic may be either interconnected or closed. The apparent porosity measures the interconnected pores and determines the permeability, or the ease with which gases and fluids seep through the ceramic component. The apparent porosity is determined by weighing the dry ceramic (Wd), then reweighing the ceramic both when it is suspended in water (Ws) and after it is removed from the water (Ww). Using units of grams and cm3:
Apparent porosity$=(W_w-W_d)/(W_w-W_s) *100$

True porosity $=(\rho\text{-}B/\rho)*100$

Where

$B=W_d/W_w - W_s$

B is the bulk density, and ρ is the true density or specific gravity of the ceramic. The bulk density is the mass of the ceramic divided by its volume.

### 2.4.3 Compressibility and Compactibility of metal powders

Compressibility and compactibility define the ability to form unsintered(green) compacts by die pressing of powders. Generally, the compressibility is quantified as the value of the compacting pressure required to ensure a specified green density of the sample. Therefore, the compressibility of the powder is an important factor in the design of pressing equipment. It is usually expressed in terms of green density. The compressibility is also measured by the compression ratio, which is the ratio of the green density to the apparent density of the powder. The compactibility is evaluated by the green strength of a powder compact and relates to the sample complexity, fragility, and the ability of the compact to be ejected from the die. The relation between the compressibility and the compactibility is complex because some factors can improve one of them to the detriment of the other. Thus, particles of a spherical shape generally have a higher compressibility. In contrast, an increase of particle surface area, which can be achieved by increasing the particle surface irregularity, usually increases the green strength. At the same time, such factors as increasing the compaction pressure and the temperature have competing effects on the compressibility and the green strength. These factors promote intense particle movement and deformation, which are the bases to improve mechanical interlocking.

### 2.5 Synthesis of silica nickel nanocomposite

Several techniques exist for producing silica-nickel nanocomposites. One approach involves separately acquiring silica and nickel and subsequently combining them to form the nanocomposite. Alternatively, silica can be synthesized independently, with nickel powder purchased separately for composite production. Another method involves synthesizing silica and then utilizing in-situ reactions within the silica matrix to produce nanoscale nickel particles, followed by composite synthesis. These methods offer diverse pathways for nanocomposite fabrication.

In this thesis report, our focus is on producing silica-nickel nanocomposites utilizing the sol-gel route, and after that it will mixed with aluminum to check the changes happed in the compacted sintered sample.

### 2.5.1 Synthesis of the silica nickel nanocomposite via solgel route

The synthesis of silica-nickel gels begins with the hydrolysis and poly-condensation of tetraethylorthosilicate (TEOS) in a water/ethanol solution under acidic conditions. In this specific process, a mixture of TEOS, water, and ethanol is prepared in a volumetric ratio of 1:1:2, respectively, as follows: Initially, a homogeneous solution is created by combining distilled water with NiCl2, along with a 100% excess of glucose compared to the stoichiometric requirement for complete reduction of $NiCl_2$, and ethanol, all under stirring. Simultaneously, another homogeneous solution containing ethanol and tetraethyl orthosilicate (TEOS) is prepared in a separate beaker with constant magnetic stirring. The first solution is then slowly added drop-wise into the second solution while stirring continuously with a magnetic stirrer, followed by the addition of a few drops of HCl as a catalyst. The resulting solution is allowed to gel at room temperature. Samples are prepared with varying weight percentages of nickel, ensuring a 100% excess of glucose beyond that required for the complete reduction of $NiCl_2$.[1]

The insitu reduction of $NiCl_2$ in silica gel occurs as follows

$C_6H_{12}O_6 = 6C + 6H_2O \uparrow$

$C + H_2O = CO\uparrow + H_2\uparrow$

$H_2 + NiCl_2 = Ni + 2HCl\uparrow$.[1]

The nanoporous silica gel matrix plays a crucial role in facilitating in situ reduction. Firstly, the nanopores confine the reduced metal to nanometric dimensions. Secondly, the simultaneous occurrence of reactions within nanopores across the entire bulk of the silica gel matrix enhances the reduction kinetics significantly. Thirdly, the entrapment of nanosized metal within the silica matrix minimizes oxidation. Characterization of the resulting nanocomposites has been performed using X-ray diffraction (XRD).[1-9]

## 2.6 Powder metallurgy

What exactly is powder metallurgy? It's a metal-forming technique where compacted metal powders are heated to temperatures just below their melting points. Although this method has been around for over a century, it's gained significant recognition in the past 25 years as an exceptional means of crafting top-notch parts for various critical applications. This rise in popularity owes to the numerous advantages it holds over alternative metal forming methods like forging and metal casting. These advantages include better material utilization, intricate shape complexity, precise control over dimensions nearing the final shape, and more. Such benefits contribute significantly to sustainability, earning powder metallurgy a well-deserved reputation as a green technology.

### 2.6.1 Powder metallurgy techniques (cold die and sintering)

Powder metallurgy (PM), although not always fully appreciated, has long been a trusted process for crafting high-quality engineering components across various industries. Essentially, PM involves blending metal powders, pressing them into molds, and then heating (sintering) the compacted powder to achieve the desired density and strength.

The historical use of powder pressing, seen in artifacts like Egyptian iron implements, the Delhi column in India, and items crafted by the Incas, underscores its longstanding presence. Commercial utilization of PM dates back to the 1920s, with the production of tungsten carbides, bronze bearings, tungsten filaments, and more. Over time, PM industries have flourished, thanks to the recognized benefits of material efficiency and component fabrication ease.

Despite its notable advantages, the powder metallurgy (PM) process does come with certain limitations. These include challenges related to part design and geometry, higher initial tooling costs, and elevated raw material expenses compared to conventional solid bulk materials. Additionally, precautions must be taken to prevent corrosion.[10-18]

While cold die compaction in powder metallurgy is generally straightforward in practice, it poses significant theoretical challenges. This difficulty likely stems from the intricate variations in parameters during the compaction process. Nonetheless, extensive research has been conducted, aiming to develop empirical and theoretical equations to describe the relationship between green density and applied pressure. The green density of compacts directly influences product densification and, consequently, its strength. However, it appears that existing expressions largely rely on experimental factors due to the presence of unknown parameters.

Efforts have been made to address these challenges, including studies involving the simulation of powder compaction using Finite Element Method (FEM). These simulations are based on the understanding of the elastic-plastic behavior of materials under large displacements, treating the powder as a continuum capable of plastic deformation when subjected to external pressure.[19-27]

After compaction sintering has to be done to increase its mechanical properties and to reduce the porosity of the samples. During the firing process, the formed piece undergoes shrinkage, leading to a decrease in porosity and an enhancement in mechanical strength. This transformation occurs through the coalescence of powder particles, resulting in a denser mass, a phenomenon commonly referred to as sintering. After pressing, many of the powder particles touch one another. In the initial stages of sintering, necks develop along the contact areas between adjacent particles. Simultaneously, a grain boundary emerges within each neck, while the gaps between particles transform into pores. With continued sintering, these pores diminish in size and adopt a more spherical shape. The primary impetus behind sintering is the reduction in total particle surface area, as surface energies outweigh grain boundary energies. Sintering temperature is usually below the melting temperature. To achieve the necessary changes, mass transport relies on atomic diffusion from the bulk particles to the neck regions.

Hot pressing involves simultaneous powder pressing and heat treatment, compacting the powder aggregate at elevated temperatures. This method suits materials that only reach a liquid phase at exceedingly high and impractical temperatures and is preferred when high densities without specific grain growth are desired. However, it's an expensive fabrication technique with drawbacks. It consumes significant time as both mold and die require heating and cooling in each cycle. Moreover, mold fabrication is costly and often results in a short lifespan.

## 2.7 Study of the processing routes of aluminum nickel composite

The use of ceramics in engineering components is limited by their brittleness. However, by integrating metallic reinforcements, the fracture toughness of ceramics has seen significant improvement[28-42]. This enhancement in toughness is attributed to the plastic deformation experienced by the ductile inclusions. For this plastic deformation to be fully effective, two conditions must be met: firstly, the elastic modulus of the metal should be lower than that of the ceramic matrix to ensure that cracks are diverted towards the metallic particles, and secondly, the metallic particles must be bonded to the brittle matrix, meaning they should remain below a critical size to prevent stress-induced cracks due to thermal expansion mismatch.

Theoretical studies [43,44]propose that the toughness improvement correlates with increased inclusion volume fraction, yield strength, and diameter. Experimental observations[44,45] suggest that some degree of debonding at the ceramic/metal interface can augment plastic deformation. The extent of debonding is heavily influenced by the interface characteristics, which are, in turn, determined by the processing method employed.

In this study, nickel inclusions are incorporated into an alumina matrix through three distinct processing methods:

1. Powder metallurgy route: In this approach, the metal and ceramic powders are blended and

then sintered together.
2. Gas reduction route: Here, a nickel-aluminate spinel is reduced in a reducing atmosphere to create an alumina/nickel composite.
3. Reaction sintering route: This method involves the formation of alumina/nickel composites through the reaction between aluminum and nickel oxide.

The study investigates the optimal processing conditions for each route and compares their efficacy in forming composite materials.

**Powder metallurgy route**

The process flow diagram is depicted in the flow chart . Alumina powders and 25.2 weight percent nickel powder were subjected to attrition milling for 4 hours using isopropanol and alumina balls as grinding media. The addition of nickel particles constituted a 13 volume percent inclusion phase for achieving fully dense composites. The particle size distribution was measured both before and after milling using Granulometer HR. Powder pellets, 2 cm in diameter and 0.5 cm in height, were then formed via cold isostatic pressing (CIP) at 200 MPa. Sintering was carried out in a box furnace at temperatures ranging from 1400°C to 1700°C for a duration of 1 hour. The heating and cooling rates were 5°C/min[28]. A graphite powder bed surrounded the powder compacts to generate a reducing atmosphere at the sintering temperature used.

**Gas reduction routes**

The process flowchart is depicted in the diagram. Alumina and 30 weight percent nickel oxide were subjected to attrition milling. The preparation procedures for the powder compacts mirrored those of the powder metallurgy route. The resulting powder compacts underwent heat treatment in air, either at 1600°C for 1 hour, 1600°C for 10 hours, or 1300°C for 10 hours. Subsequently, the compacts were sintered in a reducing atmosphere for 1 hour at either 1600°C or 1700°C[28]. Additionally, some powder compacts that were not heat-treated were sintered simultaneously.

**Reaction Sintering Route**

The process involved 14.4 weight percent aluminum droplets and 29.0 weight percent nickel oxide. The reaction, $2Al + 3NiO = Al_2O_3 + 3Ni$, occurred during firing. Polished surfaces were achieved by cutting the samples along the axial direction of the discs and then polishing with diamond paste to a 1-micron finish.

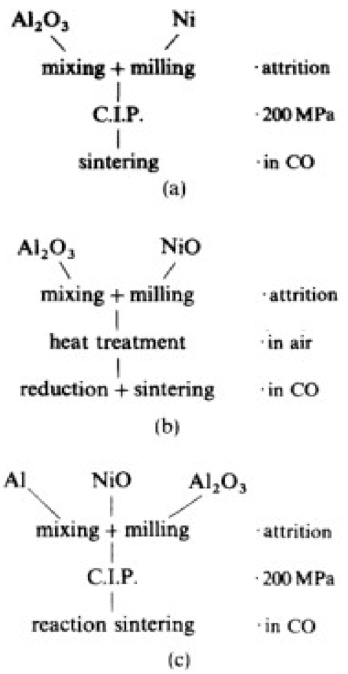

**Figure** 2.2 : Flow chart-The flow diagrams for the processing routes investigated in the present study: (a) powder metallurgy route, (b) gas reduction route, and (c) reaction sintering route.

## 2.8 Study of aluminum nickel ferrite composite

Ferrites and ceramic magnetic materials represent a crucial category within magnetic materials. Ongoing research on ferrites has yielded significant advancements, including innovative theories, models, and manufacturing techniques. Ferrites find widespread applications in motors, generators, transformers, sensors, and telecommunication due to their exceptional magnetic properties, cost-effectiveness, and ease of use. These properties stem from ferromagnetic elements like Fe, Co, and Ni.[46,47]

Nickel ferrite ($FeNi_2O_4$) stands out as a ceramic compound with a spinel structure, possessing favorable chemical and magnetic properties even at high temperatures. Its applications span across electrical communication components, sensors, drug delivery systems, and catalysts.[48-50]

On the other hand, aluminum, being paramagnetic, exhibits limited magnetic properties in strong magnetic fields. However, when combined with magnetic ceramic reinforcements, aluminum-based composites offer advantages over traditional magnetic materials, such as higher ductility, lower density, and easier shaping processes.[51]

The integration of magnetic ceramic reinforcements into paramagnetic metallic alloys to create metal matrix composites (MMCs) with distinct magnetomechanical properties remains relatively unexplored in the literature.[52] While some studies have experimented with adding magnetic particles like Fe3O4 to aluminum matrices, comprehensive analysis of the resulting composite's magnetic properties is lacking.

Metal matrix composites (MMCs), comprising metallic alloys reinforced with ceramic materials, have garnered considerable attention for their ability to enhance various properties.[53-55] Lightweight alloys such as Al, Ti, and Mg are commonly used as matrices for MMCs. Aluminum matrix composites (AMCs), in particular, are gaining prominence due to their unique blend of characteristics, including light weight, high modulus, strength, hardness, controlled thermal expansion coefficient, thermal stability, corrosion resistance, abrasion resistance, wear resistance, and excellent damping capabilities.[56-58] AMCs are also used in aerospace, automotive, sport, marine and electronics [59–65].

So far, a diverse range of ceramic materials, including oxides, carbides, nitrides, and borides, have been utilized to reinforce aluminum matrices, aiming to enhance various properties[66,67]. However, among these options, SiC and $Al_2O_3$ emerge as the most frequently employed reinforcements[68]. Incorporating ceramic reinforcements contributes to a reduction in the density of metal matrix composites (MMCs)[66], while simultaneously improving their mechanical, electrical, chemical, and physical characteristics through the addition and dispersion of these reinforcements within the matrix[68]. The effectiveness of these enhancements depends on several factors, such as the manufacturing process, type and quantity of constituents, morphology, distribution, and size of the reinforcing phase in the matrix, as well as the microstructure and the presence of defects, notably pores and cracks. Additionally, the quality of the interfacial bonding between the reinforcement and the matrix[62,66,69] significantly influences these properties. However, undesirable interfacial compounds [66]may form due to reactions between the matrix and the reinforcement. Conversely, employing thermodynamically stable materials for reinforcement, which resist phase transformation or reaction with the matrix at elevated temperatures, is crucial for ensuring the efficient performance of MMCs in high-temperature environments.[68]

It's been demonstrated that reducing the size of reinforcement particles from micron to nanometer scale increases the density of barriers against dislocation motion within the matrix, thus enhancing mechanical properties. However, overly fine particle sizes may result in excessive agglomeration and uneven distribution within the matrix.[51,53,69]

Composite fabrication methods fall into four main categories: solid, liquid, semisolid, and vapor state routes. Among these, the powder metallurgy route is a solid-state process involving blending, ball milling, compaction, and sintering of meticulously chosen powders. Advantages of this method include constituent homogeneity, desirable mechanical properties, near-net shape dimensions, cost-effectiveness, and high potential for automation and industrialization.[70,71,72]

Consequently, incorporating nickel ferrite nanoparticles into an aluminum matrix holds promise for creating a lightweight metal matrix composite with suitable magnetic properties for diverse industries such as automotive, aerospace, and precision measurement instruments.

### Density of aluminum nickel ferrite composite

The mechanical properties of powder metallurgy products are influenced by their bulk density. Various process parameters, including the density of the green body, gas entrapment during fabrication, sintering time and temperature, and the particle size of the initial powders, dictate the porosity content and distribution in a composite. Experimental measurements of sample density can be conducted using Archimedes' principle, while theoretical estimations can be made using the rule of mixtures hence, the relative density and porosity of samples can be calculated.. The theoretical density of $NiFe_2O_4$ is 5.38 g/$cm^3$[3]. The relative density of composites decreases and porosity increases with the rise in weight percent of reinforcement. Since nanoparticles tend to agglomerate, increasing the nickel Ferrite nanoparticle content in the matrix directly impacts agglomeration tendency and, consequently, the formation of defects such as porosity. Moreover, agglomerates of ceramic nanoparticles negatively affect the intergranular connection between aluminum particles. This diminishes the likelihood of diffusion during sintering, hindering densification. With the increase in reinforcement content, non-uniformities in the microstructure are observed. This uneven distribution of reinforcement particles may result from nanoparticle agglomeration and clustering, leading to porosity formation and subsequent density decline. Thus, despite the density of constituents, nanocomposites typically exhibit lower densities.[46]

### Microstructure of aluminum nickel ferrite composite

Figure 2.3 exhibits the FESEM micrographs of aluminum matrix nanocomposites reinforced with 1, 2.5, 5, and 10 wt% of $NiFe_2O_4$ after extrusion. It is evident that with an increase in the reinforcement content, more particles are dispersed within the matrix. In comparison to other samples, not only particles but also agglomerates of $NiFe_2O_4$ are noticeable in the composite containing 10% reinforcement. Moreover, the higher reinforcement content leads to the formation of more pores in the microstructure, as clearly depicted in the micrographs of 5% and 10% $NiFe_2O_4$-reinforced composites. Although augmenting the content of hard ceramic particles can improve mechanical properties, the presence of agglomerates and pores can adversely impact them. Hence, the reinforcement content must be optimized to attain the desired properties.

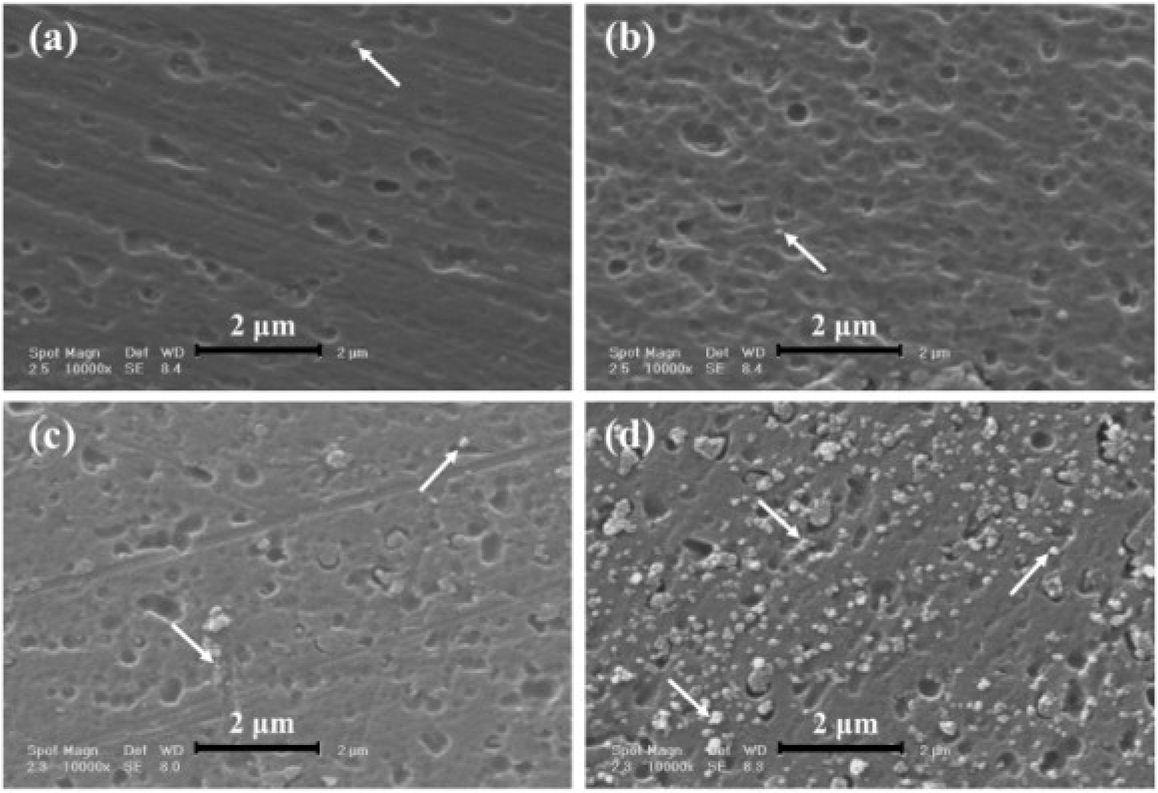

**Figure** 2.3 :FESEM Micrographs of extruded Al-$NiFe_2O_4$nanocomposites containing a) 1%, b) 2.5%, c) 5%, and d) 10% reinforcement. Arrows indicate the $NiFe_2O_4$ nanoparticles

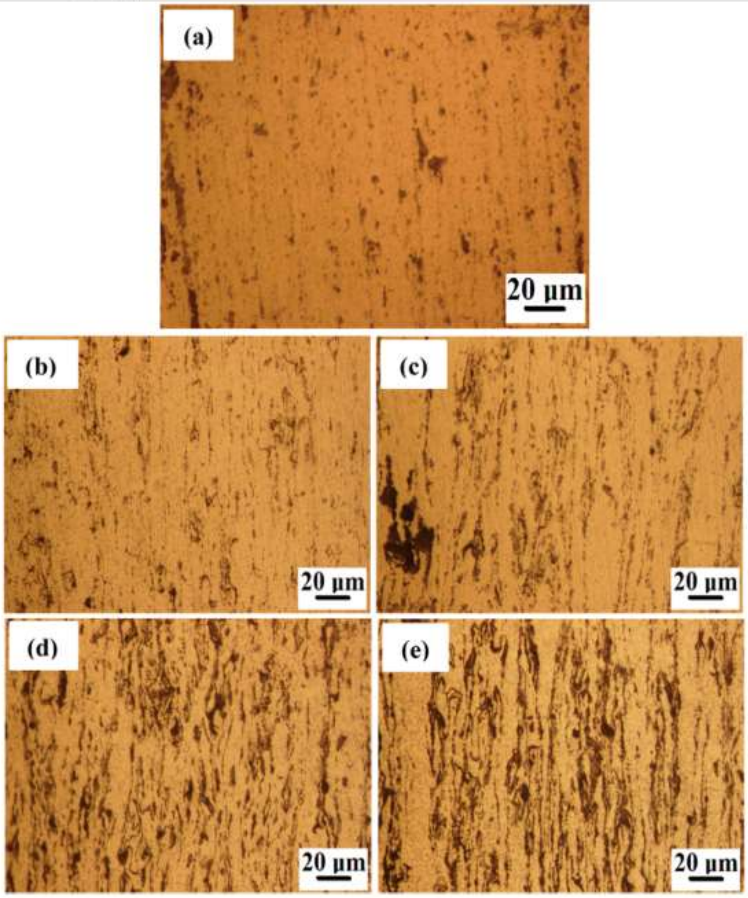

**Figure** 2.4 : Optical micrographs of a) 0, b) 1, c) 2.5, d) 5 and e) 10 wt% nickel ferrite nanoparticle reinforced aluminum matrix composites.

### Mechanical property of aluminum nickel ferrite composite.

Mechanical properties of the nanocomposites were evaluated through tensile, compression, and hardness tests. It's evident that increasing the reinforcement content up to 5wt% improves the yield stress and tensile strength of the samples. However, these properties slightly decline for the 10 wt% reinforcement composite compared to the 5wt% reinforcement sample. Additionally, elongation of the composite samples decreases compared to the monolithic sample.

According to the Orowan strengthening mechanism, the uniform distribution of nanoparticles at the grain boundaries and within the grains could enhance yield stress and ultimate tensile strength. Generally, nanoparticles enhance sample strength by impeding dislocations and increasing resistance to plastic deformation. However, agglomeration can weaken this effect, leading to a decline in strength. Therefore, there exists an optimal reinforcement content beyond which tensile properties degrade. In the Al-$NiFe_2O_4$ nanocomposite, when the reinforcement content reaches 10wt%, nanoparticle agglomeration occurs, forming large clusters with some porosity. This weakens the bond between the ceramic nanoparticles of the reinforcement phase and the matrix, while also decreasing interparticulate connection among aluminum powder particles in the matrix, leading to poor sintering and larger pores.

The nanocomposite reinforced with 10wt% nickel ferrite exhibits 25% higher strength compared to pure aluminum, with strength enhancement in other samples falling between those of the monolithic sample and the 10 wt% nickel ferrite-reinforced composite[46]. Notably, in compression tests, pores have the least adverse effect on mechanical properties, thus increasing reinforcement content continuously enhances compressive strength.

Regarding hardness, it's observed that hardness increases with increased reinforcement content, with the maximum achieved hardness being 42% higher than that of pure aluminum[46]. This is attributed to the presence of hard ceramic reinforcement particles, which elevate the hardness of the composites.

### Magnetic properties of aluminum nickel ferrite composite.

Incorporating $NiFe_2O_4$ as a magnetic component into the paramagnetic aluminum matrix alters the magnetic characteristics of the resulting composite. The magnetic attributes of composites reinforced with nickel-ferrite nanoparticles are influenced by various factors, both inherent and external, such as particle distribution, size, shape, purity, and interparticle interactions. These factors can either diminish or enhance the magnetic properties.

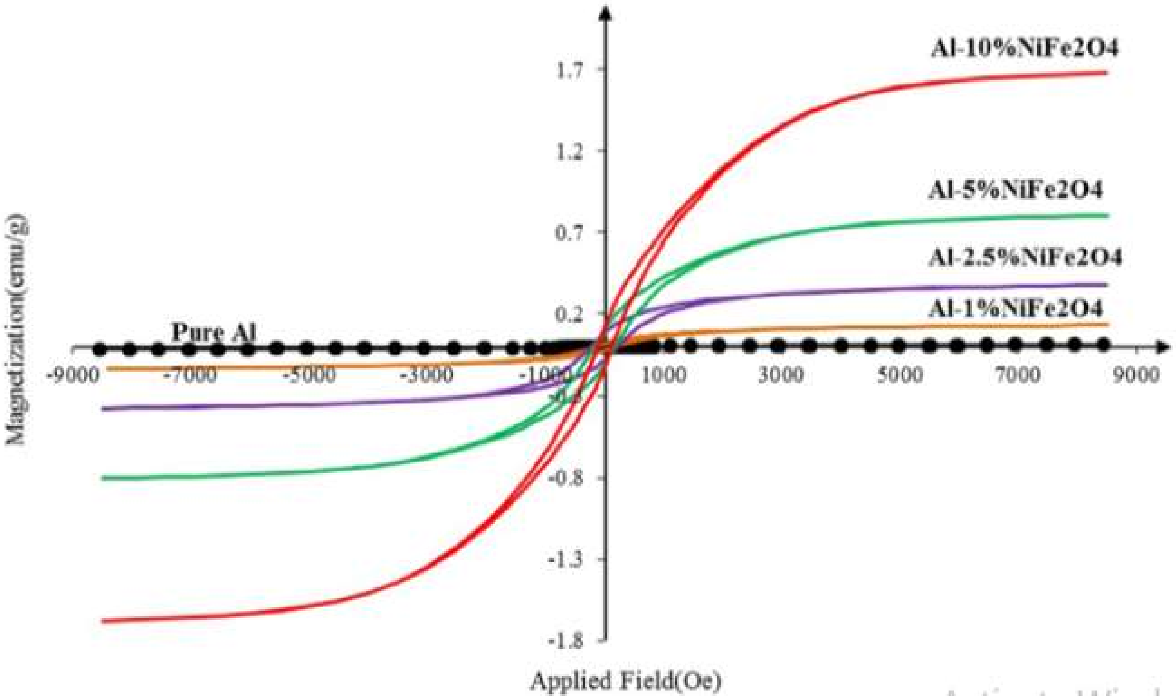

**Figure** 2.5 : Hysteresis loop (VSM graph) of pure aluminum and aluminum composites reinforced with 1, 2.5, 5 and 10wt% nickel ferrite.

Fig. 2.5 depicts the hysteresis loops acquired through VSM testing of both monolithic and nanocomposite samples[3]. The figure illustrates that both the saturation magnetization (Ms) and coercivity (Hc) increase with the rise in weight percentage of nickel ferrite ($NiFe_2O_4$) nanoparticles as the magnetic phase in the nanocomposite. While the magnetic properties of pure aluminum are negligible, the magnetization and coercivity of the nanocomposite sample, reinforced with 10wt% of magnetic nanoparticles, are measured at 1.7 emu/g and 121 Oe, respectively. This enhancement is attributed to the increased number of magnetic dipoles and their alignment along the applied field. Furthermore, the magnetic anisotropy of composites escalates with the ferrite nanoparticle content, leading to an enhancement in magnetic properties.

VSM results reveal that the saturation magnetization limit for the aluminum matrix composite, reinforced with nickel ferrite, is 1.7 emu/g, significantly lower than the Ms for pure bulk nickel ferrite. This disparity arises because aluminum, being a non-magnetic material, disrupts uniformity and diminishes magnetic properties. Additionally, the magnetization of nanoparticles is notably lower than that of bulk material due to their larger specific surface area to volume ratio[46].

## 2.9 Study of nickel particulate-reinforced aluminum composites produced by hot pressing.

Aluminum and its alloys are prized for their combination of light weight, affordability, eco-friendliness, and performance. By incorporating reinforcements, aluminum matrix composites (AMCs) can surpass these attributes. AMCs enhance properties like strength, stiffness, thermal expansion coefficient, wear resistance, damping capacity, and high-temperature performance.

While continuously reinforced AMCs (using continuous fibers) offer advantages, such as improved properties, they suffer from complex processing, limited fabricability, and high costs. Conversely, discontinuously reinforced AMCs (utilizing whiskers, short fibers, and particulates) boast simpler manufacturing processes, lower costs, and potentially isotropic properties.

In recent years, extensive research has focused on AMCs reinforced with ceramics, exploit ting their benefits in strength and modulus enhancement. However, the reactivity of aluminum with most metals limited the exploration of metallic reinforcements due to the formation of brittle intermetallic compounds. Yet, metallic reinforcements offer the advantage of improving strength without sacrificing ductility, alongside potential cost benefits compared to ceramics.

Nickel's high strength and elastic modulus make it an appealing reinforcement for aluminum alloys, while intermetallics, particularly aluminum-nickel (Al-Ni) intermetallics, show promise due to their high hardness and strength. However, the brittleness of certain Al-Ni intermetallic compounds, as indicated in the Al-Ni phase diagram, poses challenges.

Studies have examined the utilization of Al-Ni intermetallics in enhancing aluminum alloys, highlighting their potential for improving mechanical properties. The presence of Al-Ni intermetallics at the interface between Ni particles and the aluminum-silicon (AlSi) matrix can significantly impact the strength and ductility of composites. Thus, a thorough characterization of this interface is necessary to understand the effect of these in situ-formed compounds on composite performance.

In composites, effective load transfer from the matrix to the reinforcement relies on a strong interfacial bond between the matrix and the reinforcing material. A weak interfacial bond can result in lower mechanical properties compared to the matrix material itself. For instance, Liu and Bathias reported such an adverse effect in two aluminum alloys reinforced with Al2O3.

While powder metallurgy (PM) processing from Al and Ni powders can lead to the formation of Al-Ni intermetallics, solid-state processes are known to minimize the reaction between the matrix and reinforcement, particularly compared to molten metal methods. PM routes offer better control over the kinetics of Al-Ni intermetallic formation.

Recent work using friction stir processing to disperse Ni particulates (Nip) in pure aluminum demonstrated an increase in ultimate tensile strength (UTS) with Ni additions, along with the formation of Al3Ni. Although effective for locally reinforcing components, this method is not suitable for uniformly and massively reinforced structural components. PM routes, on the other hand, are better suited for such purposes, offering advantages like near-net-shape processing and the ability to produce geometrically complex components.

Despite limited studies on PM routes (press and sintering or hot pressing), they have been conducted using pure aluminum as the matrix. In some instances, Ni is combined with SiC as reinforcement, while in others, sole composites with Nip content are evaluated for their thermal and mechanical properties.

This study represents pioneering work, providing an assessment of ultimate tensile strength (UTS) and elongation to rupture of three Nip-reinforced AlSi matrix composites obtained through hot pressing. Additionally, it evaluates the influence of Nip additions by comparing AlSi-Ni composites with AlSi alone, correlating Nip content with the mechanical properties of the composites.

## Mechanical testing

Tensile tests were performed at room temperature (23 degree C), with a crosshead speed of

0.02mm/s, in a servohydraulic machine (Instron 8874), equipped with
a 25 kN capacity load cell. The results are the average values obtained from five tensile tests.

**Tensile property**

All composites exhibit higher tensile strength compared to the AlSi matrix. The AlSi-20wt%Ni composite demonstrates the most significant improvement in strength among the composites, with an 18.4% increase, while the AlSi-5wt%Ni composite achieves an 11.1% increase. However, in terms of ductility, measured by the percentage of elongation to rupture, all composites show lower values than the matrix.

These results can be attributed to several factors:

(i) During cooling from the sintering temperature, the difference in the coefficient of thermal expansion (CTE) between the reinforcement and the matrix leads to the formation of dislocations in the matrix, known to enhance composite strength. While Ni has a CTE of 13.3 *10^6/K, AlSi exhibits a value of 24.0*10^6/K, and the $Al_3Ni$ intermetallic has a value of 14.3*10^6/K. Consequently, the disparity in CTE values is more pronounced between $Al_3Ni$ and AlSi than between $Al_3Ni$ and Ni, resulting in residual compressive stress preferentially forming between AlSi and the $Al_3Ni$ interface.

(ii) The hardening of the AlSi matrix occurs due to dislocation motion blocking, potentially leading to dislocation entanglement, which strengthens the matrix during plastic deformation caused by the presence of $Al_3Ni$/Ni and $Al_3Ni$ particulates.

(iii) The load transfer effect from the AlSi matrix to the $Al_3Ni$/Ni and $Al_3Ni$ particulates is significant. These reinforcements possess higher elastic modulus values (Ni: 204 GPa, $Al_3Ni$: between 116 and 152 GPa) and higher ultimate tensile strengths (Ni: 462 MPa, Al3Ni: around 2160 MPa) compared to the AlSi matrix (typically with a Young’s modulus of 70 GPa and ultimate tensile strength of 164.56 MPa).

**Hardness measurements**

Hardness measurements were performed on samples of AlSi, AlSi-5wt%Ni, AlSi-12.5wt%Ni, and AlSi-20wt%Ni. A DuraScan model EMCO-TEST hardness tester was utilized, with a dwell time of 15 seconds under a load of 200 grams-force, to determine Vickers Hardness.

The AlSi-Ni composites exhibited higher hardness values than pure AlSi. This can be attributed to the presence of both Ni and Al3Ni phases in these composites, as discussed earlier. Nickel has a hardness of approximately 115 HV, while $Al_3Ni$ is reported to have a hardness of 841 HV.
To assess the extent of the formed interface (Al3Ni) in the AlSi-Ni composites, five FESEM images were analyzed for each composite..

The AlSi-5wt%Ni composite exhibited 10.5% of the area occupied by Al3Ni intermetallics, whereas in the AlSi-20wt%Ni composite, this value increased to 35.3%. Regarding hardness, the addition of 5wt%Ni resulted in a 16.6% increase in hardness, while with 20wt%Ni, the increase was approximately 47%. These findings demonstrate a strong correlation between the content of Al3Ni intermetallics (as assessed by the interface area fraction) and the observed increase in hardness.

# CHAPTER 3

## *OBJECTIVE OF PRESENT WORK*

In material engineering field our main focus is to study about the material and understand its nature for manipulating its behavior in various situations and use them as our own convenience. In our modern world we are surrounded with the materials so, if we can understand the behavior of the materials properly then we can make our life more simple and we can live our life more effectively. In this materialistic world material science or material engineering is a emerging topic research and advancement, and in this topic composite is one of the most important and interesting topic. A composite material (also called a composition material or shortened to composite, which is the common name) is a material which is produced from two or more constituent materials. These constituent materials have notably dissimilar chemical or physical properties and are merged to create a material with properties unlike the individual elements. Within the finished structure, the individual elements remain separate and distinct, distinguishing composites from mixtures and solid solutions. There are different kind of composite like conventional composite hybrid composite, nanocomposite etc. In this thesis topic our main focus is in nanocomposite which can be called hybrid composite. Actually we are trying to entrapped the magnetic property of ferromagnetic material mainly nickel which is nano form in the aluminum matrix composite with the help of silica. In this way we can entrap the magnetic property of nickel in aluminum and use that composite as a soft magnetic material. Main objectives for this thesis is

- Synthesis of nanosize nickel particle through solgel techniques via reduction of silica nickel chloride gel.
- Development of hybrid composites utilizing synthesized nickel and silica into aluminium.
- The another aim is to examine how the porosity and hardness of the samples change with varying weight percentages of nickel in the aluminum samples.
- Entrapped the magnetic properties of nickel in aluminum and use this property to meet the industrial and societal needs.

# Chapter-4

## *Experimental work and methodology*

## 4.1 Material used in the synthesis process

### 4.1.2 What is Tetraethyl orthosilicate?

Tetraethyl orthosilicate (TEOS), also known as tetraethoxysilane, is a chemical compound with the formula $Si(OC_2H_5)_4$. It is a colorless liquid with a sharp odor. TEOS is derived from orthosilicic acid and ethanol.

**Uses of Tetraethyl orthosilicate**

- TEOS is a precursor in the production of silica (silicon dioxide, $SiO_2$). It is commonly used in the sol-gel process, where it undergoes hydrolysis and condensation reactions to form silica nanoparticles or thin films. These silica materials find applications in coatings, optical devices, and ceramics.
- TEOS is utilized to produce thin films of silicon dioxide on various substrates, such as glass, ceramics, and semiconductor wafers. These coatings provide protection against corrosion, abrasion, and environmental degradation. They are also used as dielectric layers in microelectronics fabrication.
- TEOS can serve as a catalyst in certain chemical reactions, particularly in the synthesis of silicon-containing compounds.
- It is used in the textile industry as a water repellent and flame retardant agent. TEOS can be applied to fabrics to improve their durability and resistance to moisture and heat.
- TEOS is incorporated into adhesives and sealants to enhance their bonding strength and resistance to environmental factors.
- TEOS is used in the synthesis of inorganic polymers, such as silsesquioxanes and silica gels. These materials have diverse applications in coatings, membranes, and biomedical devices.
- TEOS-derived silica materials are investigated for various biomedical applications, including drug delivery systems, tissue engineering scaffolds, and bioimaging agents. The biocompatibility and tunable properties of silica make it suitable for these applications.

- TEOS is a versatile chemical with widespread applications in materials science, electronics, textiles, and biotechnology. Its ability to form silica-based materials with tailored properties makes it valuable in numerous industries.

Figure 4.1 : TEOS containing container

### 4.1.3 What is Nickel Chloride?

Nickel chloride, also known as nickel(II) chloride or just nickel chloride, is a chemical compound with the formula $NiCl_2$. It is a green crystalline solid that is highly soluble in water. Nickel chloride can exist in several hydrate forms, such as the hexahydrate $NiCl_2 \cdot 6H_2O$, which is the most common form encountered in commerce..

**Uses Of Nickel Chloride**

- Nickel chloride is commonly used in electroplating processes to apply a layer of nickel onto a metal surface. This process enhances the metal's corrosion resistance, wear resistance, and aesthetic appeal.

- It serves as a catalyst in various chemical reactions, particularly in organic synthesis. Nickel chloride catalysts are employed in processes such as hydrogenation and polymerization.
- Nickel chloride is used in a variety of chemical reactions as a precursor or catalyst. For example, it's used in the production of other nickel compounds, such as nickel oxide and nickel hydroxide..
- Nickel chloride is used in the manufacture of rechargeable nickel-cadmium (NiCd) batteries and nickel-metal hydride (NiMH) batteries.
- It's utilized as a reagent in various laboratory procedures, particularly in coordination chemistry experiments.
- In certain applications, such as in the purification of solvents, nickel chloride can act as a desiccant, absorbing water from the surroundings.

**Figure** 4.2 : Nickel chloride hexahydrate containing container

### 4.1.4 What is Aluminum?

Aluminum, denoted by the symbol Al with atomic number 13, is a chemical element. Compared to other common metals, it boasts a lower density, approximately one-third that of steel. Upon exposure to air, it readily forms a protective oxide layer, showcasing a visual resemblance to silver and sharing its exceptional light-reflective properties. Aluminum is characterized by its softness, nonmagnetic nature, and ductility. With its primary stable isotope, 27Al, being notably abundant, aluminum ranks as the twelfth most prevalent element in the universe. Additionally, the radioactivity of 26Al finds application in radiometric dating.

Aluminum, recognized for its silver and white hue, is a lightweight metal. It is highly malleable and plentiful in nature, commonly found in igneous rocks or as a component in gemstones such as topaz and garnet. While aluminum doesn't occur naturally in its metallic form, it can be synthesized through the combination of oxygen or other elements.

**Uses of aluminum**

- Aluminum offers numerous advantages over other metals commonly utilized by consumers and production companies. Its durability and malleability render it indispensable in various global industries, facilitating the creation of machinery, heat conduction, and the manufacturing of containers and household goods. Several key properties distinguish aluminum from its metal counterparts:

- Lightweight: Aluminum's lower weight compared to metals like copper, steel, or iron makes it a preferred choice in transportation and aerospace applications.

- Strength: Exhibiting durability in diverse weather conditions, including extreme temperatures, aluminum maintains its structural integrity even under extensive use.

- Corrosion Resistance: Despite scratches or dents, aluminum remains robust, further enhanced by protective coatings. Unlike many metals, it does not rust.

- Electrical Conductivity: With superior electrical conductivity relative to metals such as iron or copper, aluminum alloys find application in products like power lines.

- Thermal Conductivity: Aluminum efficiently conducts heat or cold, enduring varying temperatures, making it a staple in engines, heaters, and refrigerator components.

- Non-Magnetic: Its non-magnetic nature allows for its usage in a wide array of electronic devices without interference.

- Reflectivity: Recognized for its reflective properties, aluminum is frequently employed in lamp reflectors and shielding against radio waves or light.

- Recyclability: Highly recyclable, aluminum undergoes rapid processing without compromising its properties or strength.

- Versatility: As one of the most malleable alloys, aluminum finds application in diverse sectors, from household items to aircraft components, owing to its malleability, durability, and aforementioned attributes.

The picture of the powder container is I figure 4.3

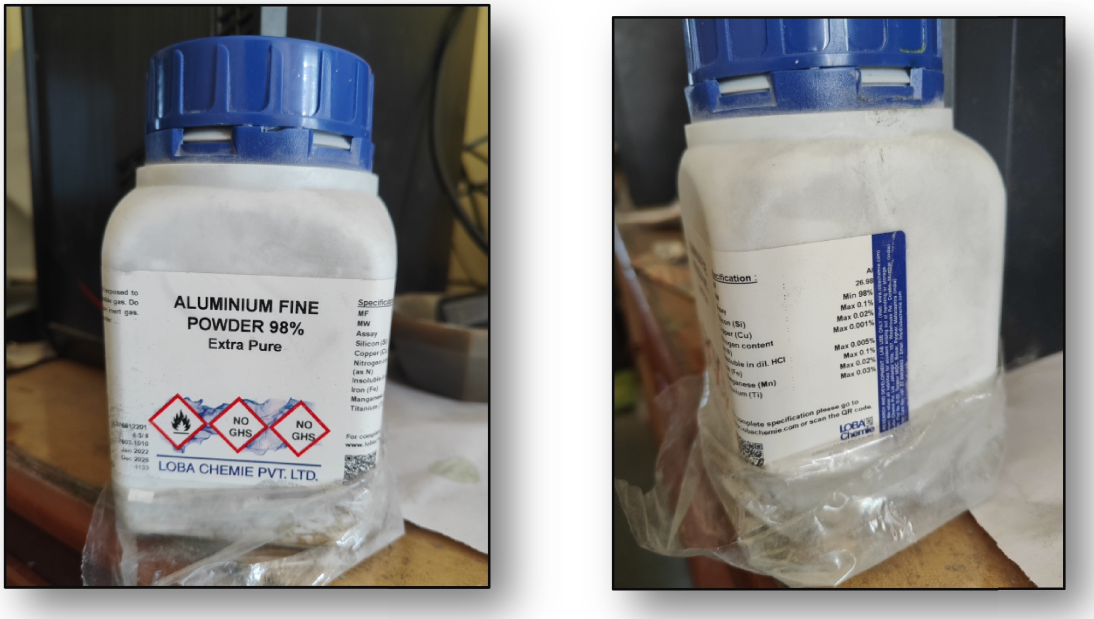

**Figure** 4.3 : Aluminum Powder containing container

### 4.1.5 What is dextrose?

Glucose or dextrose, with the chemical formula $C_6H_{12}O_6$, stands as a paramount sugar in nature. It reigns supreme among monosaccharides, a vital subgroup within carbohydrates. This essential molecule is primarily synthesized by plants and many algae through photosynthesis, utilizing sunlight, water, and carbon dioxide. Its pivotal role extends to the formation of cellulose, the predominant carbohydrate constituting cell walls worldwide.

The term "dextrose" finds frequent usage in clinical and nutritional contexts, pertaining to patients' health and dietary intake, respectively. Conversely, "glucose" is employed in biological and physiological discussions concerning chemical processes and molecular interactions. Despite this distinction, both terms denote the same molecule, specifically D-glucose.

Dextrose monohydrate represents the hydrated form of D-glucose, featuring an additional water molecule attached. Its chemical composition is denoted by $C_6H_{12}O_6 \cdot H_2O$. Also known as hydrated D-glucose, dextrose monohydrate is commonly derived from plant starches. It serves as the predominant form of dextrose in various food applications, including beverage mixes, serving as a widely utilized glucose supplement in food production. Particularly prevalent in North America, it is often consumed as corn syrup or high-fructose corn syrup.

Anhydrous dextrose, in contrast, refers to glucose devoid of any water molecules. The process of obtaining anhydrous substances typically involves the removal of water from a hydrated compound, often achieved through methods such as heating or desiccation.

In an industrial setting, dextrose monohydrate can be transformed into anhydrous dextrose through dehydration. Dextrose monohydrate contains approximately 9.5% water by mass, which is eliminated during dehydration to yield anhydrous, or dry, dextrose.

With a chemical formula of C6H12O6 and lacking water molecules, anhydrous dextrose shares the same composition as glucose. However, in open air, it has a tendency to absorb moisture and revert to its monohydrate form, making it more costly to produce.

Despite its higher production costs, anhydrous dextrose offers enhanced stability and an extended shelf life. It finds applications in various medical contexts, including oral glucose tolerance tests (OGTT), owing to its increased stability and reliability.
The picture of dextrorse powder containing container is in figure 4.4,

**Figure** 4.4 **:** Dextrose powder containing container

## 4.2 Experimental methods

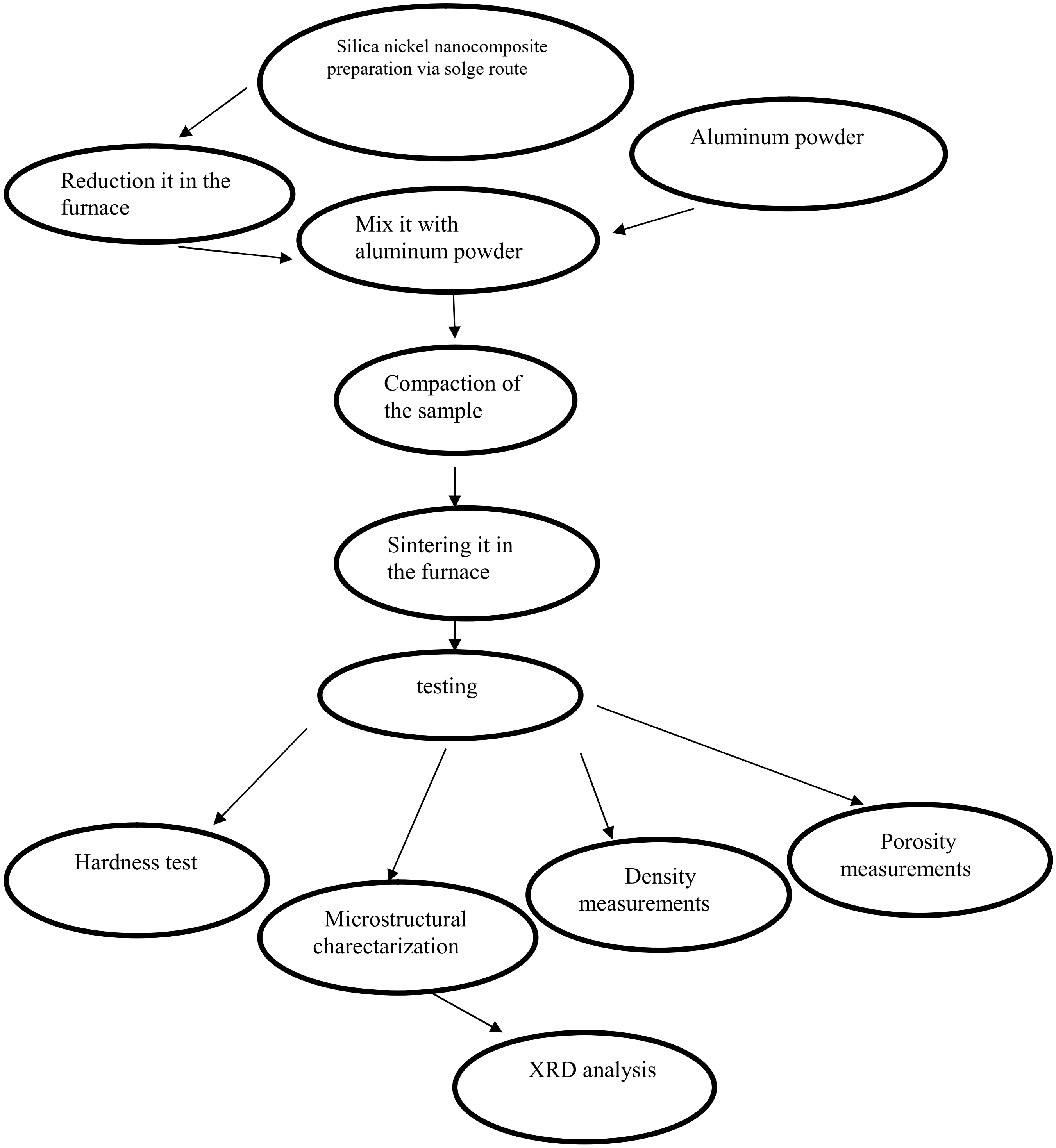

**Figure** 4.5: flow chart of experimental methods

### 4.2.1 Sample preparation

The different weight percentage of samples were prepared by powder metallurgy route.For making different weight percentage sample firstly we have synthesis the silica nickel nanocomposite via sol gel route the we have to reduce it in the furnace at specific time to silica nickel and then we had to add aluminum powder then we have to compact the sample with die pressing and after that we have to sintered the sample at specific temperature within specific time.

### 4.2.2.1 Silica nickel nanocomposite preparation

The synthesis of silica-nickel gels commences with the hydrolysis and poly-condensation of tetraethylorthosilicate (TEOS) within an acidic aqueous-ethanol medium. In this particular procedure, a solution comprising TEOS, water, and ethanol is meticulously formulated in a volumetric ratio of 1:1:2, respectively. Initially, a uniform solution is achieved by blending distilled water with $NiCl_2$ and an excess of glucose(dextrose), surpassing the stoichiometric requirement for complete reduction of NiCl2, all under agitation. Simultaneously, another homogenous solution comprising ethanol and TEOS is meticulously prepared in a separate vessel with continuous magnetic stirring. The first solution is then slowly added drop-wise into the second solution while stirring continuously with a magnetic stirrer. After 4 to 5 days the gel is formed. The solution was left the gel for 2 to 3 days to dry.
here nickel chloride hexahydrate,tetraethyl orthosilicate,,ethanol,dextrose and water was used. Two bekars ,dropper and magneto stirrer was used.

### 4.2.1.2 Reduction

After the gel was formed and dried, it was placed in the furnace for reduction. The reduction was performed at 750° to 800° Celsius. The dried gel was placed in a mortar and broken into several pieces, after which it was placed into a boat. The boat was then placed into the furnace, which had already been preheated to 750° Celsius. The boat was kept in the furnace for an average of 20 minutes. For reduction, the boat had to be placed for a minimum of 15 minutes at a minimum of 750° Celsius.Here we use electric muffle furnace for reduction

| | | | |
|---|---|---|---|
| Name | Electric muffle furnace tube type | Area | 75*75*1500 mm |
| Make | Naskar Electrical Equipments | Maximum operating Temperature | 850°C |
| Voltage | 230 V/Single phase 50 Hz/3.5 kW | | |

**Fig** 4.6 : Electric Muffle Furnace

**Table** 4.1 : Specification of electric muffle furnace

#### 4.2.1.3 Mixing with aluminum

After reduction in the furnace, the reduced silica nickel sample was placed in a mortar and ground to a fine powder. After that, aluminum powder was mixed with the previously ground powder, and a homogeneous powder was formed.

#### 4.2.1.4 Compaction

After the powders were mixed, it had to be compacted to make samples. For compaction, die pressing was used.

#### 4.2.1.5 Sintering

After compaction, sintering was a very important step. For sintering, the compacted sample was placed in the boat and put into the furnace at 650° Celsius for more than an hour. After that, it was cooled in the furnace. Once successfully cooled, it was taken out.

Here pit furnace was for better sintering,

| Name | Electric Pit furnace Temperature controlled | Area | 75*75*300 mm |
|---|---|---|---|
| Make | Multispan | Maximum operating Temperature | 1000° C |
| Voltage | 230 V/ Single phase 50 Hz/3.5 kW | | |

**Fig** 4.7 : Electrical Pit furnace Temperature **Table** 4.2 : Specification of Electrical Pit furnace

## 4.3 Determination of mechanical property

### 4.3.1 Hardness

In materials science, hardness, the opposite of softness, signifies the capacity to resist localized plastic deformation, like indentation (over an area) or linear scratching, typically induced by mechanical pressure or abrasion. Varied materials exhibit distinct levels of hardness; for instance, hard metals like titanium and beryllium surpass softer ones such as sodium, metallic tin, wood, and common plastics. Macroscopic hardness often results from robust intermolecular bonds, yet the response of solid materials to external force is intricate. Consequently, hardness can be gauged through diverse methods like scratch hardness, indentation hardness, and rebound hardness. It hinges on factors like ductility, elastic stiffness, plasticity, strain, strength, toughness, viscoelasticity, and viscosity. Ceramics, concrete, select metals, and superhard materials are common examples of hard substances, contrasting with soft matter. Hardness is a characteristic of a material, not a fundamental physical property. It is defined as the resistance to indentation, and it is determined by measuring the permanent depth of the indentation. More simply put, when using a fixed force (load) and a given indenter, the smaller the indentation, the harder the material. Indentation hardness value is obtained by measuring the depth or the area of the indentation.there is many type of hardness test like Vickers, Rockwell, Brinell, Mohs, Shore and Knoop. Which one to apply depends on the type of material to be tested and the equipment available. In our thesis we made sample of aluminum nickel silica,there is many black spot so according to the avalibility of test machine and convenience our most suitable test is micro Vickers. In micro vickers hardness test pyramid shaped diamond intendetion is used there we use 25gf of loads.

Here we use the formula, $\frac{1.854*F}{d^2}$ (F=applied load in kgf, d is the indenter diameter)

The picture of the micro vickers harndness testing machine is attached below,

**Figure** 4.8 : Micro Vicker's Hrdness Testing Machine

### 4.3.2 Density

Density is another important property for material . for powder sample bulk density is much more important than normal density.

$B=W_d/W_w - W_s$

B is the bulk density, and ρ is the true density or specific gravity of the ceramic. The bulk density is the mass of the ceramic divided by its volume.

Density of aluminum powder and the different weight percentage aluminum nickel silica sample has been taken.The density of the samples are calculated before and after the sintering process.

### 4.3.3 Porosity

Porosity is another important property for material. Porosity refers to the extent to which a material contains empty spaces or voids within its structure. These voids, known as pores, can vary in size, shape, and distribution. Porosity is a critical property in various fields, including materials science, geology, and engineering, as it influences the material's characteristics such as density, strength, permeability, and thermal and electrical conductivity.

Understanding porosity is crucial for assessing the performance and suitability of materials for specific applications. High porosity can weaken mechanical properties, reduce thermal or electrical conductivity, and affect material durability. Conversely, controlled porosity can impart desirable properties such as improved insulation, increased surface area for reactions, or enhanced fluid permeability.

The apparent porosity is determined by weighing the dry ceramic (Wd), then reweighing the ceramic both when it is suspended in water (Ws) and after it is removed from the water (Ww). Using units of grams and cm3:

Apparent porosity=$(W_w-W_d)/(W_w-W_s)*100$

True porosity =$(\rho-B/\rho)*100$

Where

$B=W_d/W_w - W_s$

B is the bulk density, and ρ is the true density or specific gravity of the ceramic. The bulk density is the mass of the ceramic divided by its volume.

In our experiment we measure apparent compacted porosity and apparent sintered porosity.

**Apparent compacted porosity**

Apparent compacted porosity is measured after compaction of the powder sample. Firstly we measure the normal weight of the sample by the use of the weight machine then we submerged the compacted sample in the water for 20 to 25 minutes after that we again measure the submerged weight of the sample and lastly we taken out the sample from the water and measure again the weight of the sample after that we calculated the porosity of the compacted sample by the formula written above.

### Apparent sintered porosity

We follow the same steps as describe earlier in the apparent compacted sample porosity measurements but now the difference is the sample is sintered in 650°C for 1.5 hour and the the whole procedure is followed.

## 4.4 Magnetic Property

Magnetic property studies investigate materials' responses to magnetic fields, focusing on characteristics like magnetization, coercivity, and magnetic susceptibility. These properties are essential in understanding ferromagnetism, paramagnetism, diamagnetism, and antiferromagnetism. Techniques such as vibrating sample magnetometry (VSM) and superconducting quantum interference device (SQUID) magnetometry are commonly used. These studies are crucial for developing applications in data storage, medical imaging, and electronic devices. They also contribute to advancements in material science, leading to the design of new magnetic materials with tailored properties.

The B-H loop, or hysteresis loop, is crucial in studying magnetic properties, graphically representing the relationship between magnetic flux density (B) and magnetic field strength (H). This loop provides insights into coercivity, showing resistance to demagnetization; remanence, indicating residual magnetization; hysteresis losses, representing energy lost per cycle; and saturation magnetization, showing the maximum magnetization a material can achieve. Understanding the B-H loop aids in designing magnetic materials for specific applications, such as transformers, inductors, and memory storage devices, by providing a comprehensive view of their magnetic behavior.

UGC DAE CSR Kolkata center helps to carry out the magnetic property test

## 4.5 Microstructural charectarisation

### 4.5.1 Microstructure study

Microstructure is crucial in material study, representing the material’s appearance under a microscope at magnifications greater than 2.5X. To study microstructure, an optical microscope is needed. This microscope, also known as a light microscope, uses visible light and lenses to produce enlarged images of small objects. It likely took its present form in the 17th century. Basic optical microscopes are straightforward, but more complex designs enhance resolution and contrast. Objects are observed on a stage through eyepieces; high-power microscopes present the same image in both eyepieces, while stereo microscopes create a 3D effect. Images, or micrographs, are often captured with a camera.

For microstructure characterization, optical microscopes reveal the basic structure and morphology of samples. When materials are sectioned, polished, and etched, their structural features become visible. These features can be seen with the naked eye or a low-powered optical microscope, while finer sub-structures require an electron microscope. Many engineering properties are structure-sensitive, meaning they relate directly to microstructural features. For example, elastic constants are structure-insensitive, varying little with composition or grain size. Conversely, properties like yield strength, thermal conductivity, and electrical resistivity are highly structure-sensitive, significantly affected by microstructural parameters such as grain size, dislocation density, and second-phase particle distribution.After completing the compaction and sintering process of the sample it under goes a meticulous sample preparation regimen in preparation for observation under an optical microscope. Initially it undergoes a polishing using various grades of polishing paper to achieve the surface smoothness. Subsequently, a cloth polishing machine (Leco spectrum system 1000) is used to get refined appearances of the samples, ensuring the elimination of any scratches or aberration. For the etching process Keller’s reagent is used. It consists of 2.5 ml of HNO3,1.5 ml of HCL, 1 ml of HF, and 95 ml of Distilled water .After preparation of the reagent the samples are subjected to etching Followed by drying, rendering them ready for examination under an optical microscope, specifically “Leica DM 2700M” microscope.

The picture of clothing machine is in figure 4.7,

**Figure** 4.9 : Clothing Machine

The picture of optical microscope setup is in figure 4.8 ,

**Figure** 4.10 : Optical Microscope Setup

### 4.5.2 XRD study

M. von Laue is acknowledged for his pivotal role in understanding the nature of X-rays, building upon their discovery by Roentgen in 1895. In 1912, Laue proposed that if mineral crystals consisted of regularly arranged atoms, potentially serving as centers for scattering X-rays, and if X-rays behaved as electromagnetic waves with wavelengths comparable to interatomic distances in crystals, then it should be feasible to diffract X-rays using crystals. Through classical experiments under Laue's direction, these hypotheses were tested, establishing the wave nature of X-rays and the periodic arrangement of atoms within crystals.

Crystalline structures exhibit a systematic and periodic organization of atoms (or ions) in a three-dimensional pattern. Due to the regular spacing of atoms, each crystal contains planes of atoms separated by a constant distance, characteristic of the crystal type.

X-rays consist of electrostatic and electromagnetic fields oscillating in periodic cycles in planes perpendicular to their direction of propagation through space. They are typically generated within evacuated X-ray tubes by bombarding a metal target (anode) with high-velocity electrons. Monochromatic X-rays used for diffraction possess specific energy and wavelength characteristics determined by the target metal. Energy transfer from high-velocity electrons to target atom electrons temporarily creates electron orbital vacancies, which are filled by electron transfers from higher energy levels. Each electron transfer results in the emission of an X-ray photon, with energies and wavelengths characteristic of the target metal.

Diffraction involves the scattering of X-rays by crystal atoms, with scattered rays reinforced in specific directions away from the crystal. This reinforcement is quantitatively related to the distance between atomic planes, as defined by Bragg's law,

$$n\lambda = 2d \sin\theta$$

Diffraction occurs whenever the Bragg law is satisfied. The wavelength of radiation remains constant for a particular X-ray tube, while the angle of incidence $\theta$ of the primary radiation with the crystal planes can be varied. X-ray spectrometers enable the variation of the angle of incidence by rotating the sample in the primary X-ray beam's path. A suitable detector intercepts and measures the diffracted rays, maintaining an angle with the sample equal to the angle of incidence of the primary beam. Direct recording X-ray spectrometers provide the value of $2\theta$ directly from their charts. The angle $\theta$ can effectively be varied through analysis with a stationary sample and recorder.

X-ray diffraction (XRD) analysis stands out as a primary tool in characterizing essential attributes like crystal structure, crystallite size, and strain, particularly in nanoparticle studies. The broadening of diffraction peaks, observed due to the random orientation of crystals in nanocrystalline materials, is a notable phenomenon. This broadening arises from the interference patterns of X-rays within a finite-sized lattice, lacking complete constructive and destructive interferences. Additionally, irregular lattice strain and structural imperfections contribute to peak broadening in XRD patterns. The size derived from the broadening of X-ray diffraction peaks provides insight into the smallest unfaulted regions or coherently scattering domains within the material. Essentially, it quantifies the size of regions bounded by defects and grain boundaries, distinguished by slight misorientations typically of one or two degrees.

Various methodologies, such as those proposed by Williamson and Hall, Warren, Averbach, and Scherrer, are available for calculating crystallite size and strain. Among these, the Scherrer equation, based on the full width at half maximum (FWHM) of a diffraction peak, is the simplest and most widely utilized for estimating the average crystallite size.

The scherrer formula is written below

$$d_{XRD} = \frac{K\,\lambda}{\beta\ COS\theta}$$

Where d is the crystallite size, the diffraction wavelength, β is the corrected Full width Half Maxima (FWHM), is the diffraction angle and K is a constant is close to unity(0.98).

The Ultima III Rigaku X-ray diffractometer (40 Kv, 30 mA) is used here, and the picture is in figure 4.9,

(NANO SC. & TECH)
Jadavpur University Met. & Mat. Engg. Department
Name of the Equipment XRD
Date of Receipt 07.09.2006
Name of the Supplier M/S. Rigaku Corpn.
From which Fund Nano Equipments & others Asstt.
Posted in Ledger Folio No. 192
Installation Date
Name of the Lab XRD LAB

**Figure** 4.11 : XRD machine

For XRD analysis we have to make the sample in powder form then place it in a holder,after that the machine is on for nearly about 30 minutes . After that we get the XRD result in the computer .there is some software present in the computer which help for further study. There is no specific important step for sample preparation but we have to make sure that the particle size is very small and we have to grind and mix it well in mortar.

## SEM study

The scanning electron microscope (SEM) stands out as a highly adaptable tool for scrutinizing and evaluating the minute structures, shapes, and chemical compositions of materials. Understanding the basic principles of light optics is crucial for grasping the fundamentals of electron microscopy. While the naked eye can discern objects at approximately 1/60˚ visual angle, translating to a resolution of around 0.1 mm (when viewed optimally at a distance of 25 cm), optical microscopy extends this resolution to approximately 2,000 Å by manipulating the visual angle through optical lenses. Optical microscopy has long been and remains pivotal in scientific inquiry. The evolution of electron microscopy emerged from the discovery in the 1890s that electrons could be diverted by magnetic fields in various experiments. This transformation involved substituting the light source with a high-energy electron beam, marking the inception of electron microscopy.

Image formation in the SEM relies on capturing signals generated from interactions between the electron beam and the specimen. These interactions can be classified into two main types: elastic interactions and inelastic interactions.

Elastic scattering occurs when the incident electrons are deflected by the atomic nucleus or outer shell electrons of similar energy. This interaction involves minimal energy loss during collision and results in a wide-angle change in the direction of the scattered electron. Incident electrons that undergo elastic scattering at angles greater than 90 degrees are termed backscattered electrons (BSE) and provide valuable signals for sample imaging.

Inelastic scattering, on the other hand, encompasses various interactions between the incident electrons and the electrons and atoms of the specimen. This results in the transfer of significant energy from the primary beam electron to the atom of the sample. The extent of energy loss depends on whether the specimen electrons are excited individually or collectively, as well as the binding energy of the electron to the atom. Consequently, the excitation of specimen electrons during the ionization of specimen atoms leads to the generation of secondary electrons (SE), typically defined as having energies less than 50 eV, which can be utilized for imaging or analyzing the sample.

Apart from the signals used for image formation, several other signals are generated when an electron beam strikes a sample, including the emission of characteristic x-rays, Auger electrons, and cathodoluminescence.

In most instances, when an incident electron collides with the surface of the specimen, it doesn't simply bounce off. Instead, these energetic electrons penetrate into the sample before encountering and colliding with a specimen atom. This process leads to the creation of a primary excitation region by the primary electron beam, from which various signals are emitted. The size and shape of this region largely depend on the energy of the electron beam and the atomic number, and consequently the density, of the specimen.. At specific accelerating voltages, the shape of the interaction volume appears as a "tear drop" for specimens with low atomic numbers and as a hemisphere for those with high atomic numbers. The volume and depth of penetration increase with higher beam energy but decrease with increasing specimen atomic number because specimens with higher atomic numbers have more particles to hinder electron penetration. One consequence of the interaction volume on signal acquisition is that using a high accelerating voltage results in greater penetration depth and a larger primary excitation region, ultimately leading to the loss of detailed surface information from the samples

In summary, the scanning electron microscope stands as an essential instrument for scientific research, engineering, quality control, and technological innovation across diverse fields, playing a pivotal role in advancing understanding and addressing real-world problems.

SEM (Scanning Electron Microscopy) and FESEM (Field Emission Scanning Electron Microscopy) are both techniques used for high-resolution imaging of sample surfaces. While SEM uses a thermionic emission source for electron generation, FESEM uses a field emission gun, providing higher resolution and improved image quality. FESEM offers enhanced surface detail and reduced sample charging compared to conventional SEM.

The field emission scanning electron microscope FESEM-FEG Inspect 50 was utilized here. The sample is prepared in powder form for obtaining FESEM images.

The picture of the FESEM is in figure 4.12

**Figure** 4.12 : FESEM setup

# CHAPTER 5

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## *Result and Discussion*

## 5.1 Composition of samples

In this experiment four samples were made of alumina silica nickel composite with different weight percentage. Actually these samples were made of different weight percentage of nickel with respect to aluminum. After reduction, the silica-nickel gel consists of 50% nickel and 50% silica. Therefore, if 4 grams of reduced silica-nickel powder are obtained from the silica-nickel gel, it contains 2 grams of nickel and 2 grams of silica. When combined with 98 grams of aluminum (which constitutes 98% of the aluminum-nickel sample in the absence of silica), the total sample composition results in 1.96% nickel and 96.08% aluminum. There is also small amount of nickel oxide present in the sample which is negligibly small in amount.

**Table 5.1 : Different weight percentage of sample used in experiment**

| Name | Sample 1 | Sample 2 | Sample 3 | Sample 4 |
|---|---|---|---|---|
| Nickel | 1.96% | 4.76% | 6.54% | 9.09% |
| Silica | 1.96% | 4.76% | 6.54% | 9.09% |
| Aluminum | 96.08% | 90.48% | 86.92% | 81.82% |

## 5.2 Density measurements

The density of the sample was measured twice: first, when the powder was compacted, and second, after the sintering was completed on the compacted samples. If a difference is observed between the compacted and sintered densities, it can be concluded that the samples become more compacted after sintering. The data for density measurements is provided below through table 5.2 to 5.3.

**Table 5.2 : Green density of the sample,**

| Sample | Mass(gm) | Diameter ($d_1$)(cm) | Diameter ($d_2$)(cm) | Average radius(($d_1$+$d_2$)/2)(cm) | Height (cm) | Density (gm/cc) |
|---|---|---|---|---|---|---|
| Al+1.96% Ni+1.96%Si | 7.63 | 1.655 | 1.654 | 0.827 | 1.634 | 2.17 |
| Al+4.76% Ni+4.76%Si | 4.96 | 1.653 | 1.655 | 0.827 | 1.114 | 2.07 |
| Al+6.54% Ni+6.54%Si | 4.52 | 1..653 | 1.655 | 0.827 | 1.020 | 2.06 |
| Al+9.09% Ni+9.09%Si | 5.25 | 1.654 | 1.653 | 0.826 | 1.195 | 2.05 |

Here it can be seen from table 5.2 that the density decreases as the weight percentage of nickel and silica increases.

**Table 5.3 : Sintered density of the sample,**

| Sample | Mass(gm) | Diameter ($d_1$)(cm) | Diameter ($d_2$)(cm) | Average radius(($d_1$+$d_2$)/2)(cm) | Height (cm) | Density (gm/cc) |
|---|---|---|---|---|---|---|
| Al+1.96% Ni+1.96%Si | 7.5 | 1.651 | 1.649 | 0.825 | 1.590 | 2.20 |
| Al+4.76% Ni+4.76%Si | 4.9 | 1.645 | 1.643 | 0.822 | 1.104 | 2.09 |
| Al+6.54% Ni+6.54%Si | 4.45 | 1.645 | 1.651 | 0.824 | 1.010 | 2.06 |
| Al+9.09% Ni+9.09%Si | 5.15 | 1.652 | 1.656 | 0.826 | 1.165 | 2.06 |

Here from the table 5.3 it can easily understood that density is decreasing for the increasing of the nickel and silica weight percentage. The density is increased after sintering implies the formation of bonding between the matrix and hybrid reinforcements.

## 5.3 Porosity measurements

Porosity was also explained earlier. It is a very important property in material science. The porosity of the different weight percentage samples was measured before and after sintering. Both apparent compacted porosity and apparent sintered porosity were calculated using universal porosity measurement technique. For the apparent compacted porosity, the powder sample was first compacted, and then a measurement setup was used where the sample was suspended via a thin thread in a beaker filled with water. The entire setup was placed on a weighing machine. The weight of the sample in its dried condition (Wd), submerged condition (Ws) after 15 minutes, and wet condition (Ww) were measured respectively.

The formula is,

Apparent compacted porosity=$(W_w-W_d)/(W_w-W_s)*100$

Similar technique is also used for apparent sintered porosity but the diference is here the sample is sintered in 650°C for 1.5 hour.

The formula here is used same as compacted porosity measurements,

Apparent sintered porosity=$(W_w-W_d)/(W_w-W_s)$ *100

The data for the above porosity calculation is give below table 5.4 ad 5.5,

**Table 5.4 : Porosity of the compacted sample**

| SAMPLE | DRY WEIGHT | SUBMERGED WEIGHT | WETTY WEIGHT | POROSITY |
|---|---|---|---|---|
| Al+1.96% Ni+1.96%Si | 7.63 gm | 6.33 gm | 7.81 gm | 11.69 |
| Al+4.76% Ni+4.76%Si | 4.96 gm | 4.48 gm | 5.13 gm | 15.17 |
| Al+6.54% Ni+6.54%Si | 4.52 gm | 3.23 gm | 4.80 gm | 17.83 |
| Al+9.09% Ni+9.09%Si | 5.25 gm | 4.09 gm | 5.56 gm | 21.08 |

By the above table it has been easily seen that the porosity is increasing with the increasing of the nickel weight percentage.

After sntering,

**Table 5.5 : Porosity of the sintered sample**

| SAMPLE | DRY WEIGHT | SUBMERGED WEIGHT | WETTY WEIGHT | POROSITY |
|---|---|---|---|---|
| Al+1.96% Ni+1.96%Si | 7.5 | 6.21 | 7.65 | 10.41 |
| Al+4.76% Ni+4.76%Si | 4.9 | 4.15 | 5.03 | 14.77 |
| Al+6.54% Ni+6.54%Si | 4.45 | 3.03 | 4.74 | 16.95 |
| Al+9.09% Ni+9.09%Si | 5.15 | 3.95 | 5.46 | 20.53 |

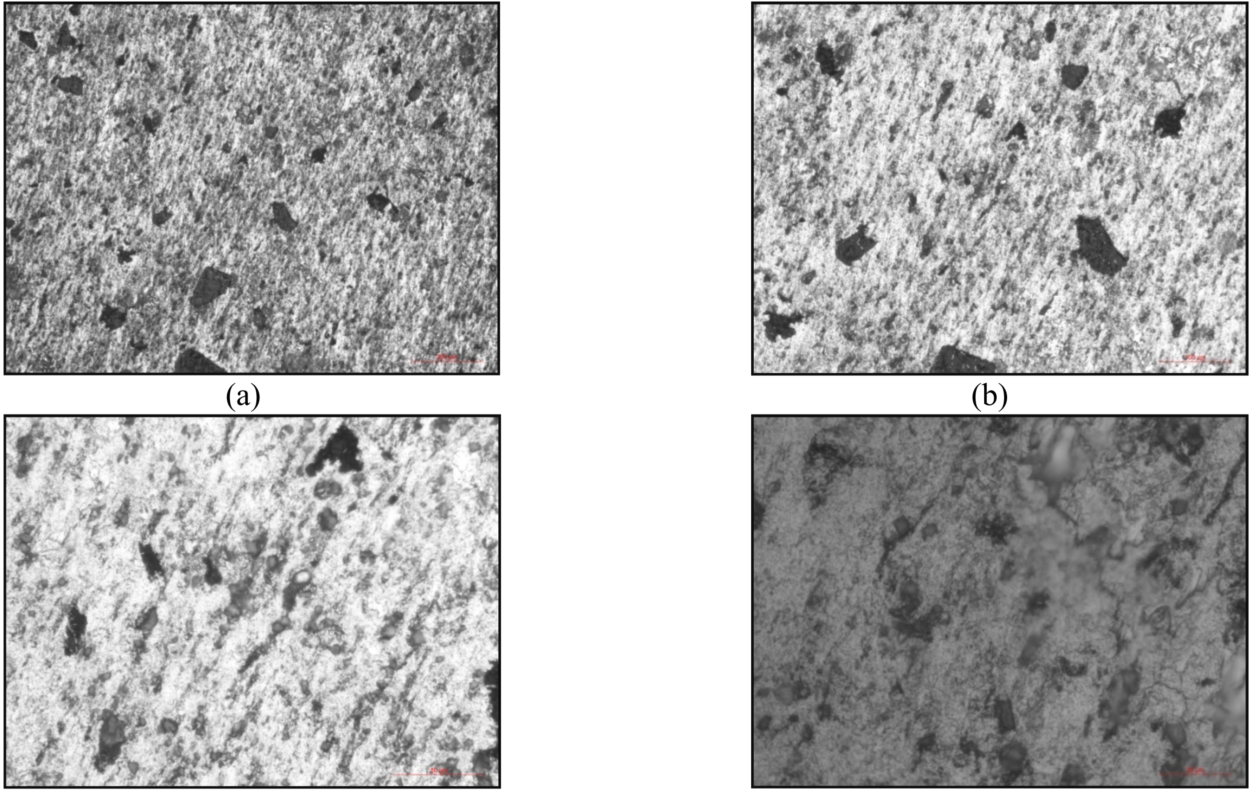
The porosity increased with the increasing nickel weight percentage in the sample. However, a comparison of the above table 5.5 also showed that the porosity decreased in the sintered sample.

## 5.4 Microstructure analysis

As discussed earlier, the microstructure is a crucial aspect of material study. Here, microstructures of four different weight percentages of the samples are taken, and the picture of this is presents in figure 5.1,5.2,5.3 and 5.4 .There is total 4 figure of microstructure of sample 1 .figure (a) ,figure (b),figure (c) and figure (d) has four has different magnification and resolution.

| Sample (1,2,3,4) | A | B | C | D |
|---|---|---|---|---|
| Magnification | 100X | 200X | 500X | 1000X |

## Sample 1

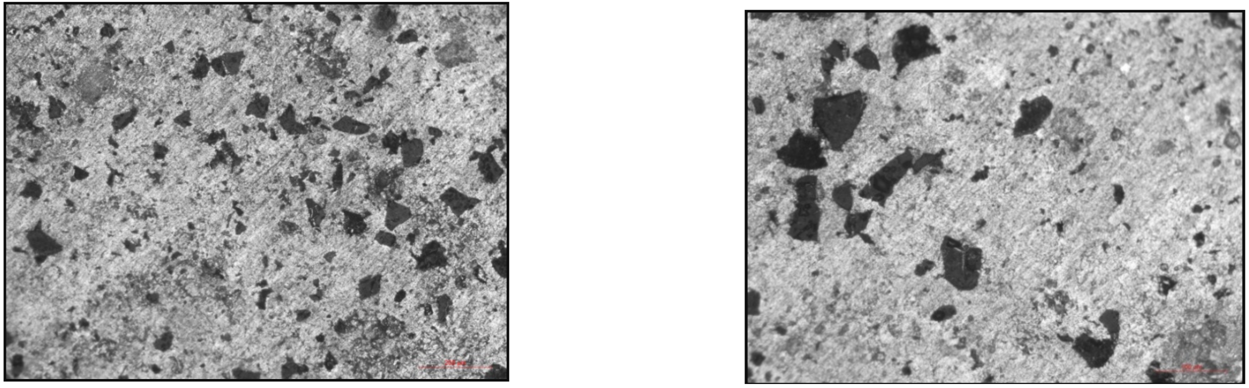


(a) (b)

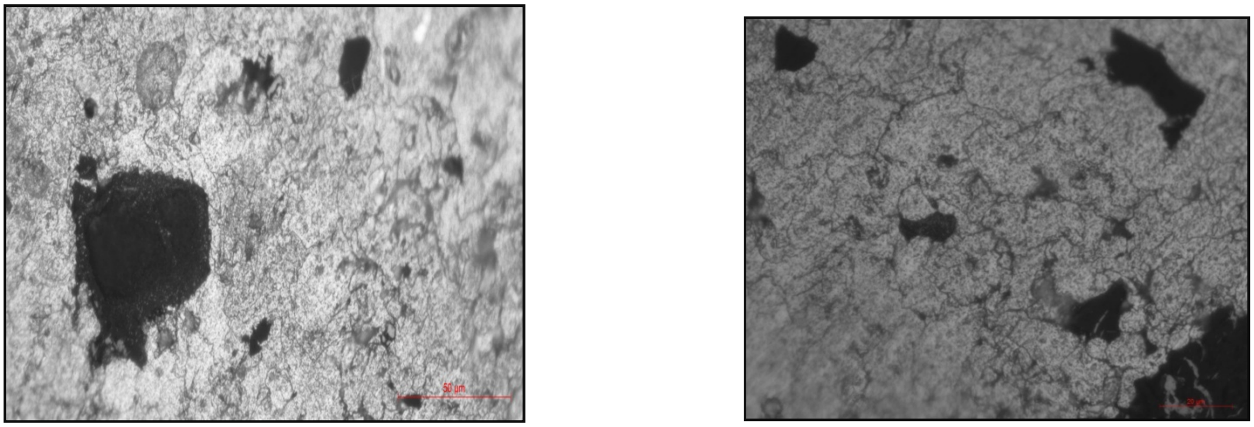
(c) (d)

**Figure** 5.1 **:** Optical micrograph of aluminum 1.96 wt % nickel 1.96wt% silica at varying magnifiaction

## Sample 2



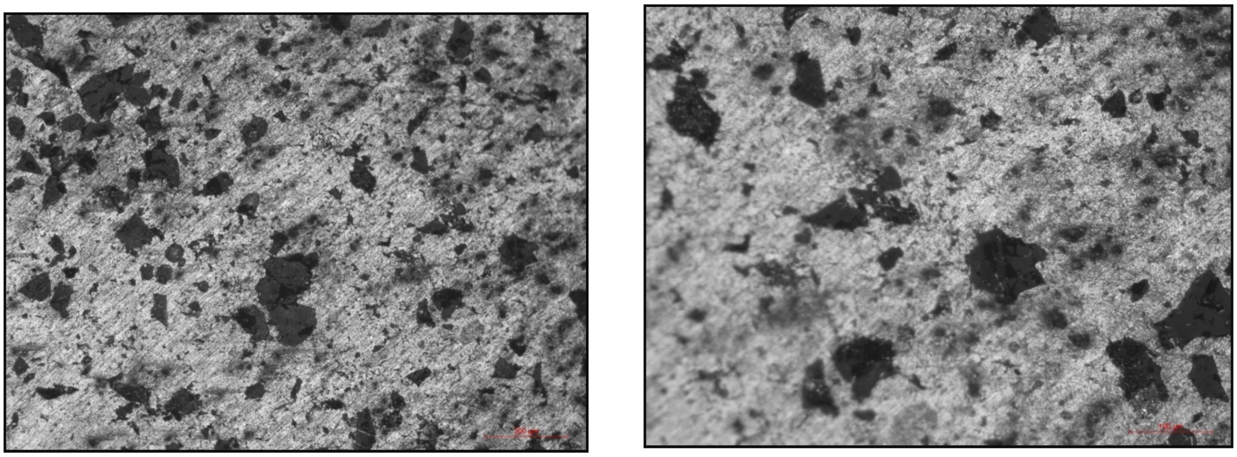
(a) (b)



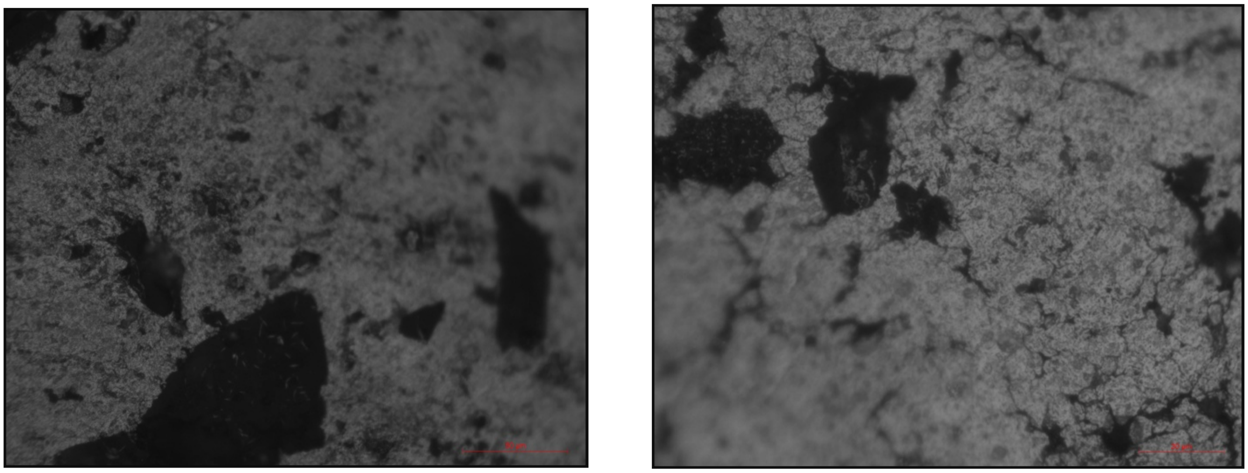
(c) (d)

**Figure** 5.2 **:** Optical micrograph of aluminum 4.76 wt % nickel 4.76wt% silica at varying magnification

## Sample 3



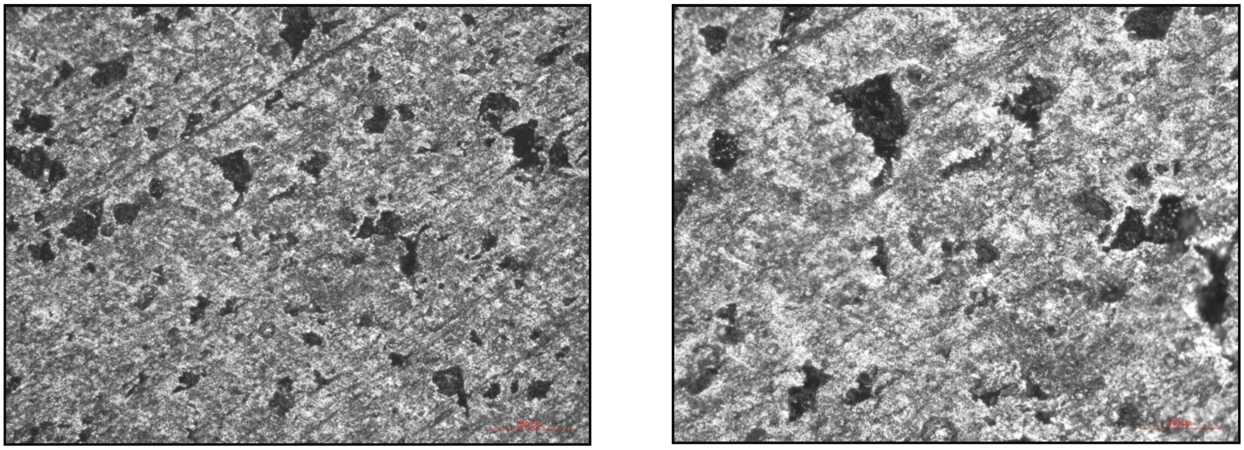
(a) (b)



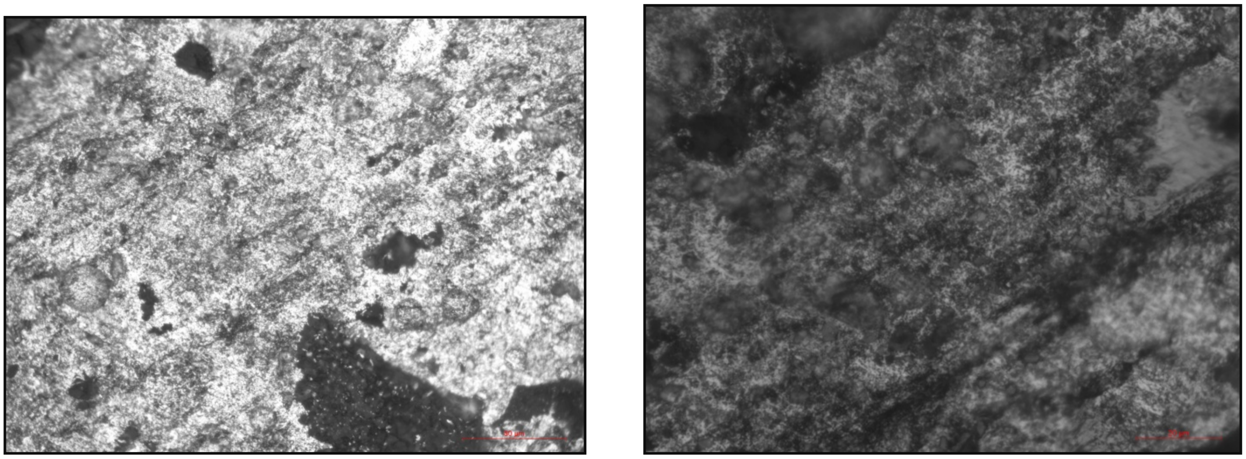
(c) (d)

**Figure** 5.3 **:** Optical micrograph of aluminum 6.54 wt % nickel 6.54 wt% silica at varying magnification

## Sample 4



(a) (b)



(c) (d)

**Figure** 5.4 **:** Optical micrograph of aluminum 9.09 wt % nickel 9.09wt% silica at varying magnification

Based on microstructure shown in fig. 5.4, it can say that the void spaces are increased with increasing the weight percentage of nano crystalline nickel and silica. This result reveals the accuracy of the porosity values.

## 5.5 Hardness analysis

The aluminum-silica-nickel nanocomposite with different weight percentages was subjected to micro hardness testing. Macro hardness survey was carried out only the optimized samples, having least porosity. Hardness was taken utilizing diamond nano indentation applying 25 gf loads.

Here, the used the micro hardness formula is

$$\frac{1.854*F}{d^2}$$

Where F is the load we used, d is the average diameter.

The data for micro hardness is in table 5.6

**Table** 5.6 : Micro hardness of aluminum nickel silica sample at different weight percentage

| | D1 | D2 | (D1+D2)/2 | HV | Average HV |
|---|---|---|---|---|---|
| Al+1.96%<br>Ni+1.96%Si | 39.8 | 39.2 | 39.5 | 29.7 | 29.2125 |
| | 41.1 | 39.1 | 40.1 | 28.9 | |
| | 39.0 | 39.6 | 39.3 | 30.0 | |
| | 40.5 | 39.6 | 40.5 | 28.25 | |

| | D1 | D2 | (D1+D2)/2 | HV | Average HV |
|---|---|---|---|---|---|
| Al+4.76%<br>Ni+4.76%Si | 38.8 | 39.4 | 39.1 | 30.31 | 31.75 |
| | 37.9 | 37.7 | 37.8 | 32.44 | |
| | 38.9 | 38.4 | 38.65 | 31.02 | |
| | 38.9 | 38.6 | 38.75 | 30.86 | |

| | D1 | D2 | (D1+D2)/2 | HV | Average HV |
|---|---|---|---|---|---|
| Al+6.54%<br>Ni+6.54%Si | 37.6 | 37.2 | 37.4 | 33.14 | 33.93 |
| | 37.5 | 36.2 | 36.7 | 34.41 | |
| | 37.1 | 36.6 | 36.85 | 34.13 | |
| | 37.0 | 36.5 | 36.75 | 34.31 | |

| | D1 | D2 | (D1+D2)/2 | HV | Average HV |
|---|---|---|---|---|---|
| Al+9.09%<br>Ni+9.09%Si | 37.8 | 37.2 | 37.5 | 32.96 | 33.43 |
| | 37.4 | 36.9 | 37.15 | 33.58 | |
| | 37.4 | 36.5 | 36.95 | 33.95 | |
| | 37.8 | 36.9 | 37.35 | 33.22 | |

As demonstrated by the table 5.6, it is evident that hardness increases with the increase in nickel and silica percentage. However, 9.09 wt % nickel sample was not as hard as 6.54 wt% nickel sample or 4.76 wt % of nickel sample , and it is also observed that 9.09 wt % sample has lower density and a more porous structure.

The graph for above hardness is in figure 5.5 ,

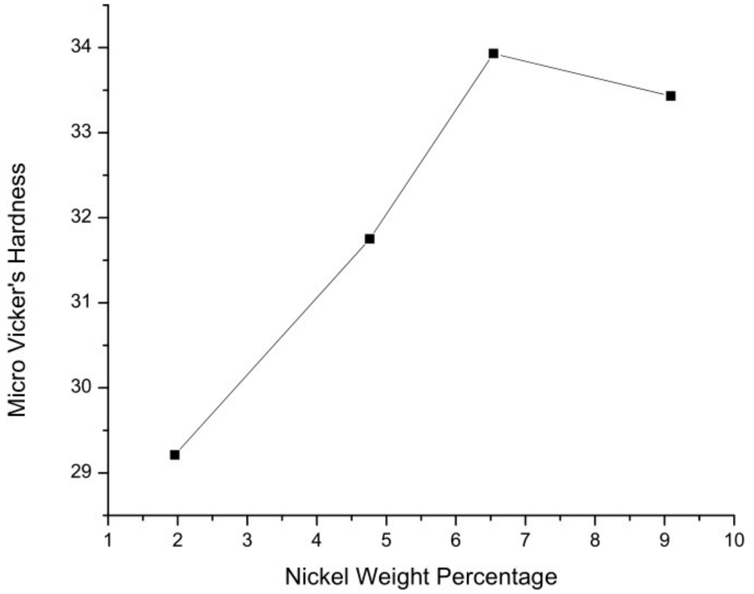


**Figure** 5.5 : Microhardness graph at variable nickel weight percentage

## 5.6 XRD analysis

X-ray diffraction patterns are widely used in nanoparticle research as a primary characterization tool for obtaining critical features such as crystal structure, crystallite size, and strain. Figure 5.6 and Figure 5.7 is the XRD pattern of the nickel silica gel before and after reduction.

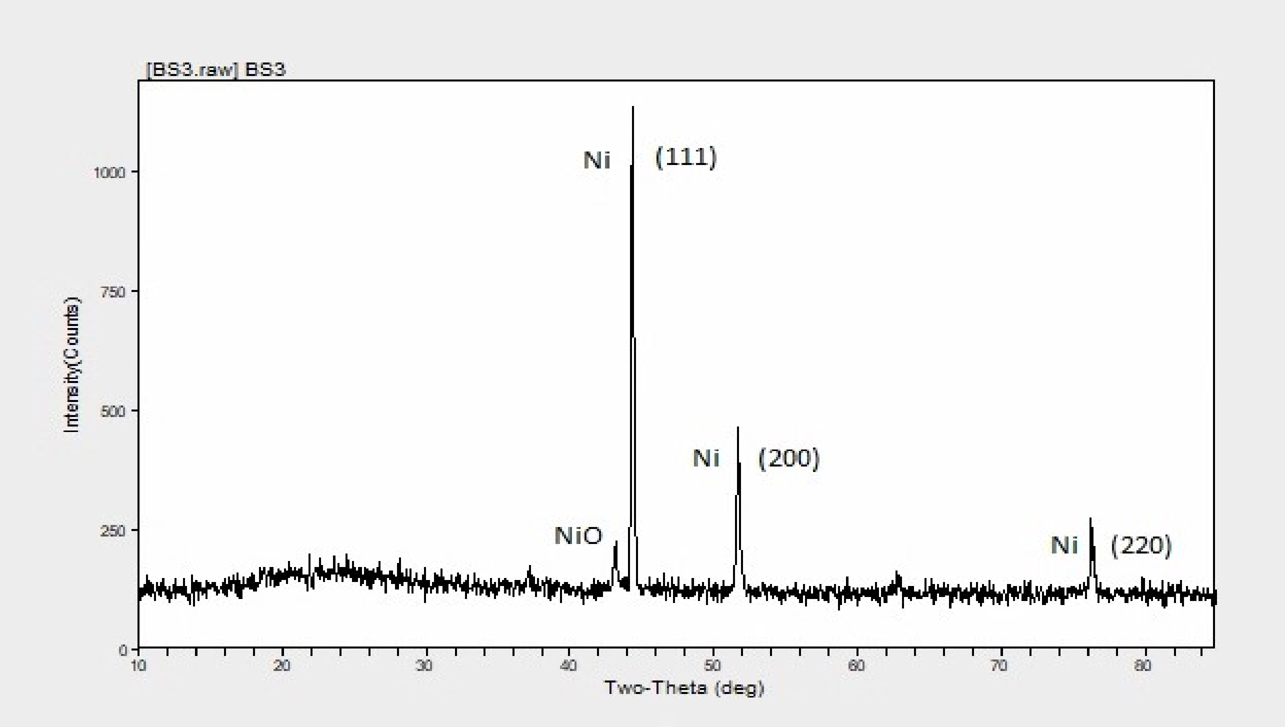


**Figure** 5.6 : XRD graph of reduced silica nickel gel

Fig 5.6 shows the X ray diff pattern of nickel silica gel , where gel is synthesized into in tube furnace at controlled environment. In the Xray diffraction pattern the presence of nickel and nickel oxide is found out and noted in the figure 5.6. Basically negligible amount of nickel oxide is shown in this pattern. Nickel has found out at the plane (111),(200) and (220).The graph has sharp peak which reveals the crystalline nature of nickel.

The fig 5.7 shows the xray diff pattern shows silica nano nickel before the reduction. No sharp peak has found out for silica gel rather it shows humped peak at two theta angle of 20° to 30°.

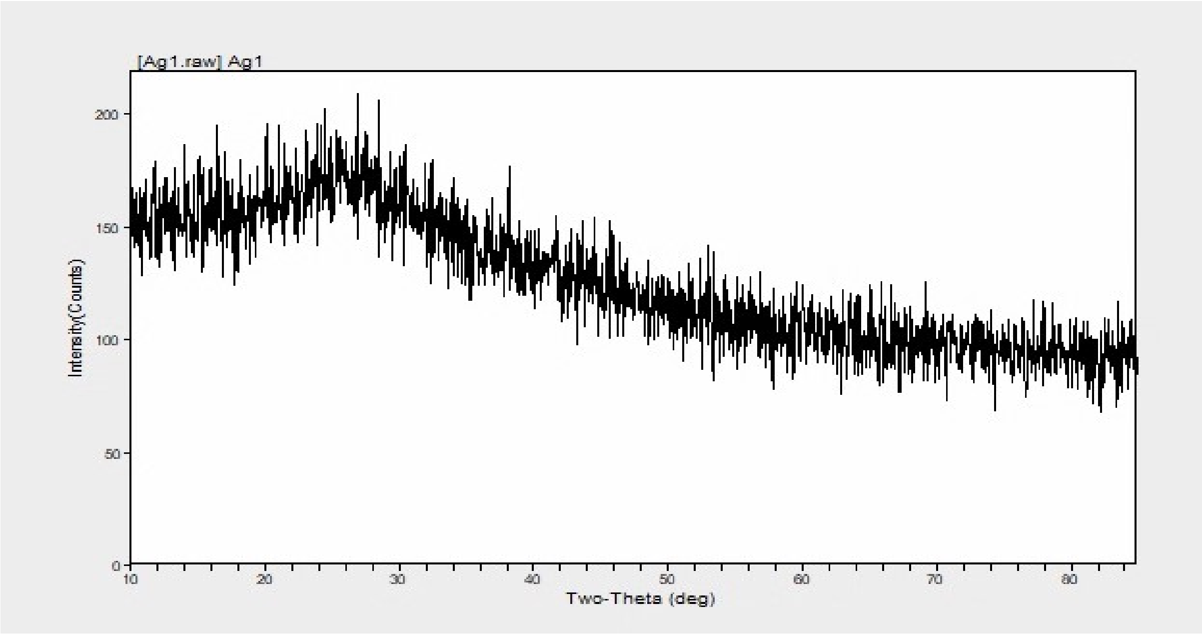


**Figure** 5.7 : XRD graph of nickel silica gel

## 5. 7 Particle size calculation

As mentioned above XRD is the one of the important tool for particle size calculations. Here we use schrrer formula for particle szie calculation.

The scherrer formula is written below

$$d_{XRD} = \frac{K\,\lambda}{\beta\,COS\theta}$$

Where d is the crystallite size, the diffraction wavelength, β is the corrected Full width Half Maxima (FWHM), is the diffraction angle and K is a constant is close to unity(0.98).

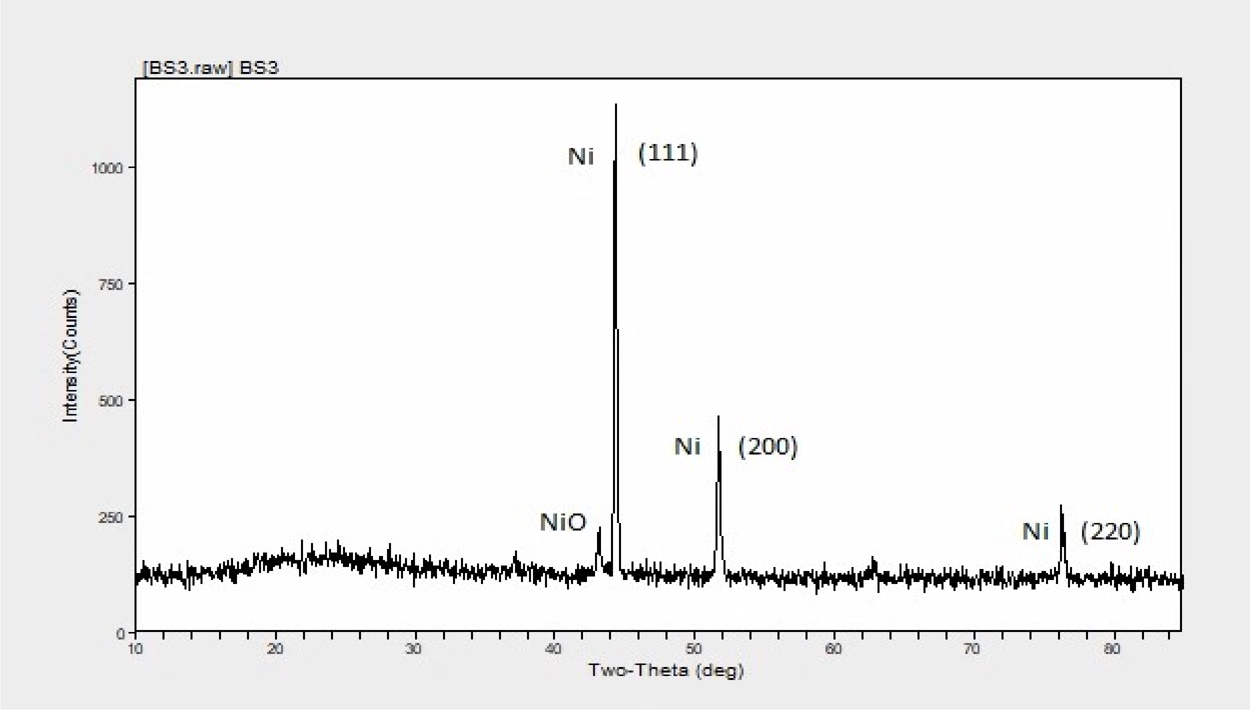


**Figure** 5.8 :XRD graph of reduced nickel silica for particle size calculation.

The XRD graph is shown in fig 5.8. Here, objective of applying scherrer equation is that whether the used synthesized nickel is nano sized or not.

**Table** 5.7 : Particle size determination

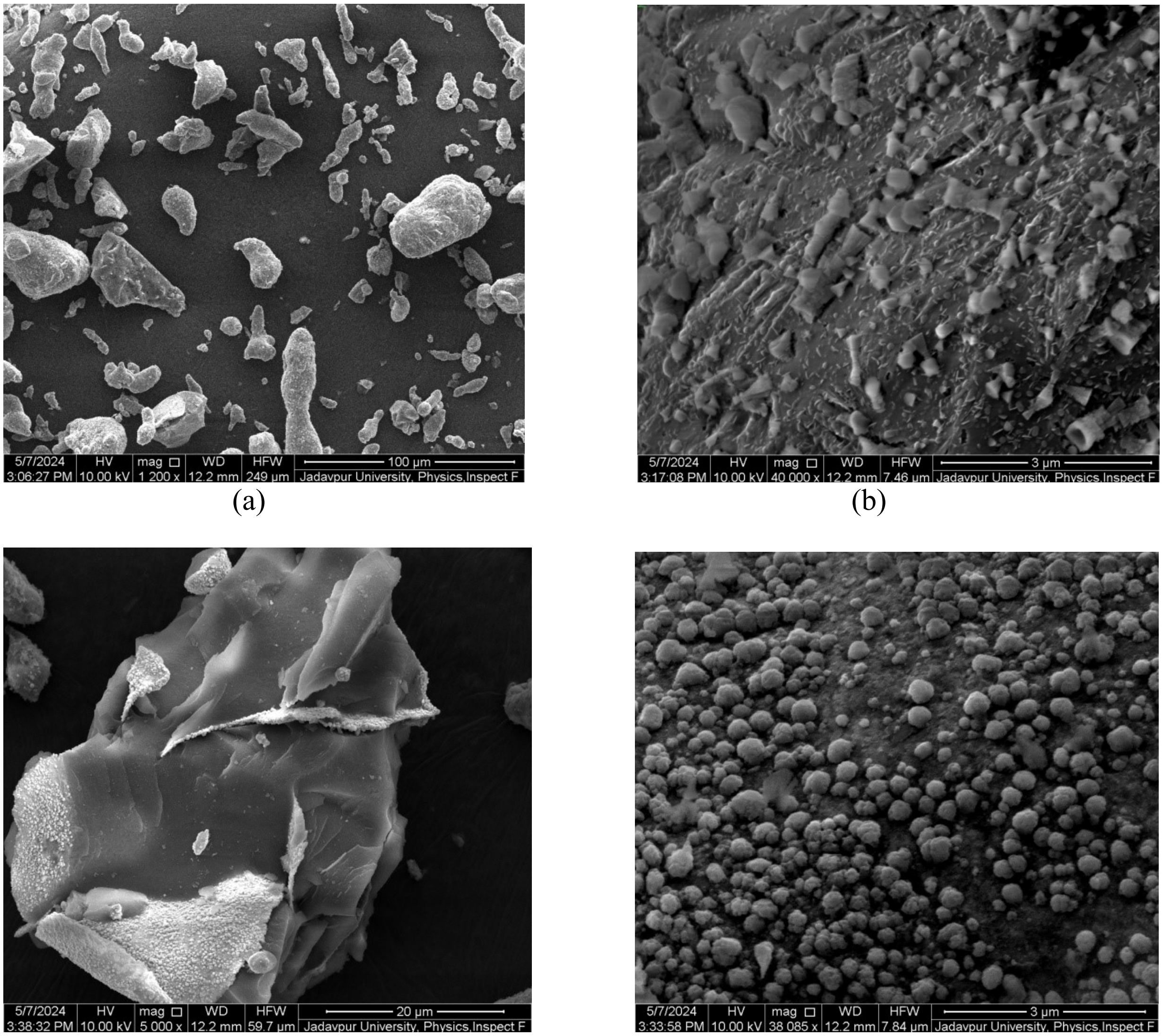
| Element | 2θ | Flex Width | d value | Particle size(nm) |
|---|---|---|---|---|
| NiO | 43.15 | 0.353 | 2.0948 | 24.21 |
| Ni(111) | 44.350 | 0.294 | 2.0408 | 29.19 |
| Ni(200) | 51.770 | 0.294 | 1.7666 | 30.05 |
| Ni(220) | 76.250 | 0.294 | 1.2477 | 34.37 |

By the use of the table 5.7 it is easily shown that we have successfully made nanoparticle.As we all know nanoparticle range is in between 1 to 100 nanometer . Here the average size is 31.20 nm.

## 5.8 FESEM analysis

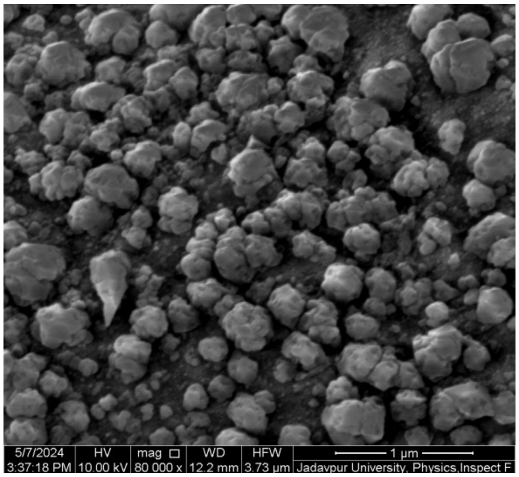
As explained earlier FESEM is a very important tool for morphology analysis . By the use of FESEM one can easily able to see the shape and structure of particles which are inside the test sample.

The FESEM images are attached in figure 5.9,



(a) (b)

(c) (d)



(e)

**Figure** 5.9 : FESEM image of aluminum 4.76 wt % nickel 4.76 wt% silica sample.

Figure 5.9 shows the surface morphology of the sample. Here, aluminum 4.76 wt% nickel and 4.76 wt% silica (sample 2) is considered for FESEM study because it has optimized properties. Nickle is prominently identified in Figure 5.9 (d) and (e). Actually studies show this type of structure is for nanoscale nickel [72 ]. Here, the shape is like close to spherical and some has also messy shape.

## Magnetic property measurements

Magnetic Property is very important for magnetic material. Figure 5.10 shows the hysteresis loop of aluminum nickel(6.54 wt%) silica(6.54 wt%) composite. Here the nickel is is nano size. By the graph magnetic saturation point and coercivity can easily calculated. It can be easily concluded from the hysteresis loop that this kind of composite can be used as soft magnetic material . From the graph represent in figure 5.10, the saturation magnetization of aluminum nickel(6.54 wt%) silica(6.54 wt%) composite was found to be 0.55 emu with a coercivity of 0.052 tesla.

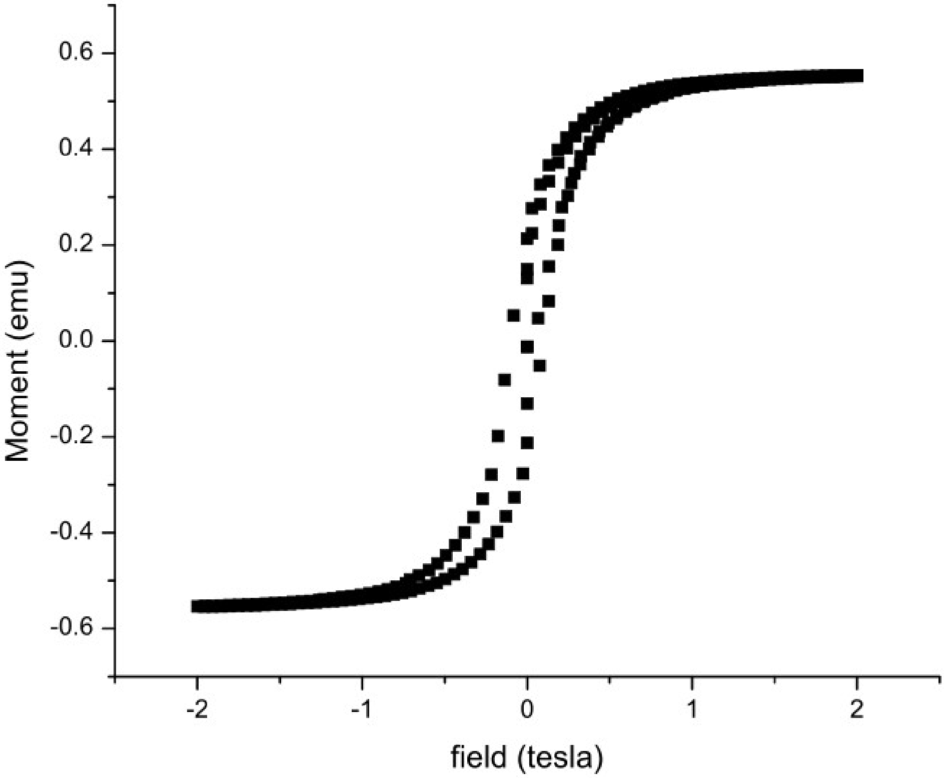


**Figure** 5.10 : Hysteresis loop of aluminum 6.54 wt% nickel 6.54 wt% silica sample.

The coercivity of nanomaterials depends on several factors, including (i) intrinsic factors, which primarily depend on magnetocrystalline surface and shape anisotropy, and (ii) extrinsic factors, generally related to interactions between particles. The interaction among nanoparticles, such as interparticle interactions, significantly influences coercivity.

So,by incorporating the nickel nanoparticle in aluminum metal matrix composite it can be seen that its magnetic property as well as some other mechanical property like hardness can also be enhanced.

# Chapter 6

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## *Conclusion and Future scope*

## CONCLUSION

Nickel-silica magnetic nanocomposite was synthesized via solge route and fabricated aluminum silca nickel composite using the powder metallurgy route. The conclusions of this study can be summarized as follows:

1. Aluminum-nickel-silica composites were fabricated with varying weight percentages: 1.96%, 4.76%, 6.54%, and 9.09%. The samples were prepared at a temperature of 650°C using powder metallurgy technique.

2. In XRD analysis the presence of nano crystalline nickel at silica are observed separately where reduction process is carried out at temperature ranging from 700° to 800°Celsius in inert atmosphere.

3. The reduced silica-nickel was mixed with aluminum powder in different weight percentages to create compacted samples, which were then sintered at 650°C for half an hour.

4. Various investigations were conducted on the samples to determine their porosity, density, and hardness. It was found that as the percentage of nickel and silica increased, the samples became less dense and more porous, with a slight increase in hardness.

5. The sample with 9.09% nickel was the least dense, most porous, and had a slightly lower hardness compared to the other samples with different weight percentages.

6. X-ray diffraction (XRD) and scanning electron microscopy (SEM) analyses were also performed on the samples to determine the size of the nickel particles, as well as the structure and morphology of the composites.

7. A aluminum-based hybrid magnetic metal matrix composite has been prepared by incorporating very low volume contents (1.96, 4.76,6.54 and 9.09 wt%) of nickel into aluminum matrix via powder metallurgy technique. The magnetic composite thus prepared exhibit excellent mechanical and magnetic properties including hardness, magnetization, and coercivity

## Future scope

Here aluminum nickel silica composite made with nano nickel . As silica is present we can see the hardness is also increased ,though it s not that much but we proper techniques and instrument we can achieved the desired result.

1. This aluminum nickel silica composite can be compacted by the use of hot isostatic press but due to time boundation in this thesis work it cannot be done. If that press can be used then the sample becomes well compacted and the desired hardness can be achieved.
2. This sample has excellent magnetic property. By the use of that property it can be used as soft magnetic material.
3. This aluminum nickel silica may be used as coating material which can increase the corrosion resistance, hardness as well as magnetic property of the sample.

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