Fabrication and Characterization of Boron Nitride Nano Sheets (BN-NS) Reinforced Aluminum Matrix Composites for Aerospace Applications

By

Abdullah Pirzada

Ibrahim Qasim

Abdul Raziq

School of Chemicals and Materials Engineering

National University of Sciences and Technology

July, 2020

Fabrication and Characterization of Boron Nitride Nano Sheets (BN-NS) Reinforced Aluminum Matrix Composites for Aerospace Applications

By

Leader – 00000175539 Abdullah Pirzada

Member 1 - 00000185743 Ibrahim Qasim

Member 2 – 00000173712 Abdul Raziq

A THESIS

Submitted to

National University of Sciences and Technology

In partial fulfilment of the requriments for degree of

B.E. MATERIALS AND METALLURGICAL ENGINEERING

School of Chemicals and Materials Engineering (SCME)

National University of Sciences and Technology (NUST)

July, 2020

CERTIFICATE

This is to clarify that work in this thesis has been completed by Mr. Abdullah Pirzada, Mr. Ibrahim Qasim and Mr. Abdul Raziq under the supervision of Dr. Malik Adeel Umer and Dr. Shoaib Butt at the School of Chemical and Materials Engineering (SCME), National University of Science and Technology, H-12, Islamabad, Pakistan.

Advisor:

Dr. Malik Adeel Umer
Department of Materials Engineering
School of Chemical and Materials Engineering
National University of Sciences and Technology

Co-Advisor:

Dr. Shoaib Butt
Department of Materials Engineering
School of Chemical and Materials Engineering
National University of Sciences and Technology

Submitted Through:

HOD----------
Dr. Zakir Hussain
Department of Materials Engineering
School of Chemical and Materials Engineering
National University of Sciences and Technology

Principal/Dean ----------
Dr. Zakir Hussain
School of Chemical and Materials Engineering
National University of Sciences and Technology

DEDICATION

This thesis is dedicated to our parents who worked hard and as a result of their hard work we reached here. Because of their love, support and prayers we were capable of completing this project and we are hopeful because of their support we will make success with leaps and bounds afterwards.

ACKNOWLEDGEMENTS

First of all we are thankful to Allah, Who is The Most Gracious and The Most Merciful. He gave us strength to tackle every problem we faced in this project and in our overall life.

Besides this we would like to show our gratitude to our supervisor Dr. Adeel Umer for his guidance and supervision. The way he explained us, tought us and corrected us in every milestone of this project was outstanding.

Last per not the least we are thankful to every class fellow who were present to help us in sorting out our problems and supporting us morally.

At the end we would like to show our gratitude to our family members who supported us morally.

ABSTRACT

The aerospace industry is desirous to develop materials with enhanced flexural, specific and compression strength. With modernization of mankind and increase in research with leaps and bounds has enhanced the existing materials to their maximum capabilities. Advancement in technology has necessitated manufacture of new materials streets ahead of existing materials.

The desire of better properties resulted in development of immense number of methodologies for constructing new materials out of which metal matrix composites are of great significance because of their high strength and ease of tailor ability depending on required application.

Often conventional aluminum alloys are mostly used in aerospace industry and are on the ball compared to other alloys. This major capture of industry is because of their high strength, low weight and cost effectiveness along with ease of recycling.

Since the aluminum alloys are already playing dominant role in aerospace industry so our research is counter-stoned on development of Al-2024 based matrix composite resulting in properties optimal for aerospace structural material..

TABLE OF CONTENTS

LIST OF FIGURES

CHAPTER 1

INTRODUCTION

1.1 History and Background

The aerospace industry is desirous to develop materials with enhanced flexural, specific and compression strength. With modernization of mankind and increase in research with leaps and bounds has enhanced the existing materials to their maximum capabilities. Advancement in technology has necessitated manufacture of new materials streets ahead of existing materials.

The desire of better properties resulted in development of immense number of methodologies for constructing new materials out of which metal matrix composites are of great significance due their high strength and ease of tailor ability depending on required application.

Often conventional aluminum alloys are mostly used in aerospace industry and are on the ball compared to other alloys. This major capture of industry is because of their high strength, low weight and cost effectiveness along with ease of recycling.

Since the aluminum alloys are already playing dominant role in aerospace industry so our research is counter-stoned on development of Al-2024 based matrix composite resulting in properties optimal for aerospace structural material.

Thus keeping in view the desired outcomes we initiate work by using Al-2024 alloy as a matrix material and hexagonal boron nitride, h-BN as reinforcement. The research is based on powder metallurgy and base materials used are in powdered form, mixing of constitutes will be done using ball milling followed by conventional pressing and finally sintered without pressure into pallets.

The first objective of the project after preparing well mixed samples is to optimize palletizing and sintering process. The goal is to find optimal conditions corresponding to maximum densification thus resulting in maximum strength properties as stated by Hall-Petch relationship.

The project has two routes depending on mixing mechanism low energy ball milling and high energy ball milling respectively. Firstly, initial powders will be mixed via low energy

ball milling followed by conventional pressing and pressure-less sintering. Secondly,

initial powders will be mixed via high energy ball milling followed by conventional pressing and pressure-less sintering in tube furnace with inert atmosphere to prevent oxidation.

Sample preparation via ball milling is in-situ process resulting in size reduction and exfoliation of boron nitride (BN) in Nano-sheets. The low energy route comprises of preparation of 4 samples having 0.1wt%BN, 0.2wt%BN, 0.4wt%BN and 1wt%BN and each weight percentage sample is prepared at three different milling times which are 24hrs, 48hrs and 72hrs respectively, thus resulting total 12 samples via low energy ball milling route. In parallel, high energy route comprises of preparation of 4 samples having 0.1wt%BN, 0.2wt%BN, 0.4wt%BN and 1wt%BN and each weight percentage sample prepared at three different milling times which are 24hrs, 48hrs and 72hrs respectively, thus resulting total 12 samples via high energy ball milling route.

The above mentioned samples are to be compared with Al-2024 alloy having 0wt%BN prepared via powder metallurgy under similar conditions. Properties which are to be evaluated and compared to pure alloy powder include flexural strength and hardness beside this percentage reduction in porosity as function of sintering time and temperature is to be evaluated and extent of BN exfoliation depending on milling time is to be compared and concluded.

Core project objectives to be achieved are listed as follow:

- In-situ fabrication of Al-2024 matrix composite reinforced with h-BN via low energy and high energy ball milling respectively
- Optimization of palletization and sintering process to achieve maximum densification and least percentage porosity
- Extent of BN exfoliation as a function of milling time
- Investigation and comparison of hardness of samples with each other and pure Al-2024 alloy
- Evaluation and comparison of flexural properties of various samples.

CHAPTER 2

LITERATURE REVIEW

2.1 Key requirments

Following are key requirements which are to be noted as these vital requirements of aerospace materials. Since the material we opt for must include the properties enlisted below:

2.1.1 High specific strenght

There are various types of polarization in the material. The most common are; orientation, ionic, and electronic. These polarizations depend on the frequency of the applied voltage.

2.1.2 High damage tolerance

The material we choose as a base of our composite must have high damage tolerance because in aerospace the material has to encounter excessive friction, tension and compression. That is why the matrix material must have high damage tolerance.

2.1.3 Low coefficient of thermal expansion

Jet fighters and aero planes experience significant rise in temperature at the points where friction is maximum, thus the material must have low coefficient in thermal expansion to avoid change in dimensions with respect to temperature.

2.1.4 High flexural strength

Another core requirement of aerospace material is to have high flexural strength to allow maximum tilt for sharp turns and efficient take-off and landing. Hence the matrix material must have high flexural strength

2.2. Powder Metallurgy

Powder metallurgy is a manufacturing technique which uses initial raw material for production in powder form. A term powder is used for any

material in granular form having particle size less than 1mm. Mostly this technique is used for metals but one might found this technique being implied to polymers and ceramics. In powder metallurgical technique, solid particles have high flow ability due to spherical shape and particles act like liquids and fill up the cavities. By utilizing flow properties, powders can be pressed to fabricate desired engineering structure or component [16].

Powder metallurgy can be divided in three pivotal stages. The first stage is formation and mixing of initial powders, second stage involves consolidation and densification of powders and finally the third stage is concerned with testing and characterization of samples to check whether desired properties are attained or not. The testing might include hardness, tensile testing, specific testing or flexural testing. Moreover, initial particle size, particle shape may influence the mixing and consolidation process.

2.2.1 Resons for powder metallurgy

Powder metallurgy is opted because of its cost effectiveness, high dimensional accuracy of product, low post manufacture machining cost [8]. As there is no melting of starting material unlike casting, casting and solidification defects are avoided deliberately so metals and alloys having high melting point can be used efficiently. This is why powder metallurgy is preferred for ceramics, high melting metals and other materials for which other processing routes are not feasible or cost effective to use.

2.3. Aluminium powder matellurgy

Powder metallurgy (P/M) has become important processing method for producing Metals parts, because of its high efficiency in moderate to high volume production of net or Near-net shapes. Current consumer products have an increasing number of P/M components and are found in automotive, aerospace, and non-vehicular applications [18]. In automotive applications, ferrous-based P/M parts predominate over all other available P/M materials. However, non-ferrous P/M components are replacing ferrous components for a number of reasons, for example:

weight reduction, equal or superior strength to weight ratios, and potential reduction of production costs. ALUMINUM P/M PARTS are used in an increasing number of applications. The business machine market currently uses the greatest variety of aluminum P/M parts. Other markets that indicate growth potential include automotive components, aerospace components, power tools, appliances, and structural parts. Due to their mechanical and physical properties, aluminum P/M alloys provide engineers with flexibility in material selection and design. Sintered aluminum P/M parts are competitive with many aluminum castings, extrusions, and screw machine products that require expensive and time consuming finishing operations. In addition, sintered aluminum P/M parts compete with other metal powder parts in applications where some of the attractive physical and mechanical properties of aluminum can be used. The following combination of properties makes aluminum attractive for P/M parts:

- Light weight
- Corrosion resistance
- High strength
- Good ductility
- Nonmagnetic properties
- Conductivity
- Machinability
- Variety of finishes

The table below shows the comparative study of various powdered alloys in terms of UTS, YS and elasticity.

Alloy	UTS (MPa)	YS (MPa)	El. (%)
Al-Fe5-Cu2-Ti2-Ce1-Zr1	357	281	3.2
Al-Fe4-Cu2-Ti1-W1-Ce1-Zr1	356	287	3.7
Al-8 Fe-7 Ce	270	225	7
Al-8 Fe-2 Mo	235	210	10
Al-12 Fe-1.2 V-2.2 Sn	310	300	7
Al-4.5 Cr-1.5 Zr-1.2 Mn	235	215	—
7075-T6 Wrought	70	55	60
2024-T81 Wrought	140	115	20

Figure 1 Properties of Extruded P/M-RS Al Alloys at 315°C

Advanced aluminum allows us to utilize advantages of rapid solidification and mechanical alloying that can be used to form new aluminum alloy. The processes of fast solidification and mechanical size reduction of powder lead to microstructural grain refinement and better mechanical properties of the alloy. Moreover, fast solidification can increase the alloying limits in aluminum by increasing super-saturation and thereby enabling greater precipitation hardening without the harmful segregation effects from over alloyed I/M alloys.

Aluminum based matrix composites are also a significant application of aluminum. Powder metallurgy of Aluminum results in production of near net shape composites with enhanced uniformity in the reinforcement distribution and relatively finer microstructure size than for other fabrication techniques thus leading to better overall properties.

2.4. Handling and Safety

The safety and handling procedures applied are an important consideration when working with aluminum powder metallurgy. Aluminum powders must be used cautiously due to their volatility, an example of their uses illustrates this as they are used as rocket fuel boosters in aluminum powered spaces shuttles.

This section will discuss the safety procedures for handling aluminum powder.

Aluminum powder suppliers should be consulted before being involved in Aluminum powder metallurgy as they are a great source for information for particular applications.

Any powder which can react with oxygen can ignite, if they are fine enough and disperse into a dust cloud, are explosion can occur. Aluminum is highly reactive and after milling its reactivity increases even more, hence its powder is considered very dangerous. The minimum ignition energy (MIE) indicates the sensitivity of any powder to ignite. Lower values of MIE indicate a higher likelihood to ignite. MIE values below 25 mJ indicate high degrees of sensitivity and can be ignited by electrostatic

charge [13]. The minimum explosive concentration determines the quantity of powder needing to be in a dust cloud to instigate an explosion upon ignition. The maximum rate of pressure rise is used when designing explosion venting. Venting prevents the buildup of pressure to explosion levels. The minimum ignition temperature is the temperature at which metal dust that's lying in a heap can ignite. Properly handled metal powders however, should never be allowed to accumulate.

For explosion to occur minimum amount of oxygen should be present in atmosphere. In nitrogen at least 9% Oxygen must be present, in helium it's 10%, while in Carbon Dioxide only 3% is needed [14]. The powder needs to be suspended, i.e. be in the form of a dust cloud for there to be an explosion.

Aluminum powder should not come in prolonged contact with water as this causes a reaction producing Hydrogen gas, thus it should be stored and handled with this in mind. This increases the hazard of using aluminum powder. Other practices which should be adopted are similar to any flammable material. Store in appropriate containers and keep away from oxidizers and combustible materials.

One should be very careful in two specific areas of aluminum powder metallurgy operations [14]. Usually metal powder is transferred from one container to another, during transferring dust clouds can formed. Make sure powder transfer is slow and deliberate. No sparking elements should be near the container.

Dust clouds can generate when powders are mixed in mixer. To prevent the formation of dust cloud inert environment should be used [14]. A mixer that reduces that reduces the creation of frictional heat is recommended.

Specific methods for cleaning metal powders should be researched and adopted. Guidelines for handling aluminum powders are available from Aluminum Association [14].

2.5. Consolidation and sintering

In powder metallurgy cold pressing (consolidation) followed by sintering is cost effective and simpler production procedure. Although the process produces near net shape products it is limited to simpler features in pressing direction yet a product can have complex features in directions other than pressing direction. For these design limitations we referred Powder Metallurgy Design Manual, published by MPIF [1]. Achieved Tolerances in cold pressing and sintering of aluminum are good. As-sintered dimensional tolerance is 0.051 mm, while the as-sized tolerance is 0.013 mm [1].In general higher the compaction pressure higher is the green density achieved. Since aluminum is having high ductility, it can be consolidated to higher densification compared to ferrous powders at relatively lower values of pressure. In order to show comparison between aluminum powder and iron powder graphs are shown below [3] [4] [20].

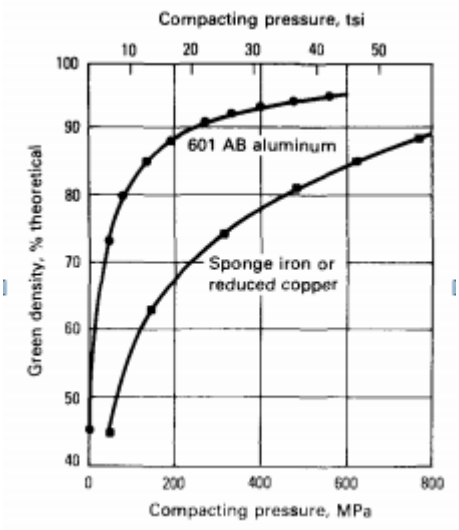

Figure 2. Relationship of green density and compacting pressure

At sufficiently high pressures aluminum powder can be consolidated to green density with zero porosity [2]. Full densification of 601AB Al was achieved at 600 MPa and that of sponge iron at 800 MPa [3] [4]. Aluminum is sintered at temperature >90% of its melting

temperature. In order to facilitate sintering process a transient liquid phase is also added. The sintering cycle usually consists of three stages. In first stage the binder is removed, the second stage consists of holding at high temperature and third stage consists of slowing cooling sample to

room temperature moreover the sintering process and resulting mechanical properties are greatly influenced by time, temperature and furnace environment [5].

For sintering of aluminum continuous and batch furnaces can be used, former provides higher production rates but later provides better atmosphere control and lower cost respectively. Moreover choice of furnace atmosphere is quite important for aluminum sintering to avoid unwanted reactions, to achieve high dimensional accuracy and mechanical properties [6]. Aluminum can be sintered in nitrogen, vacuum, inert gas and dissociated ammonia. Use of hydrogen is not appreciated due to lower properties achieved of sintered part. Argon is preferred for sintering because of higher sintering density achieved [7]. The outer porous layer reduces oxygen partial pressure in regions deep inside compacted powder thus reducing chances of oxidation.

2.6. Aluminium matrix composites

Powder metallurgy is one of the most effective fabrication technique for aluminum alloy matrix because of lower cost and better mechanical properties obtained [8]. By utilizing powder metallurgy higher weight percentage of reinforcements can be added resulting in higher tensile strength, flexural strength and hardness [15]. The very first sintered aluminum powder produces resulted in formation of Al2O3 which enhanced the strength at higher temperatures along with good thermal, electrical and corrosion resistance [9] [10]. Reaction milling can be used to increase the strength of aluminum by addition of carbon black. This can be done by heating at 270°C for 100hrs without much reduction in ductility [11].

Moreover, incorporation of nitrogen in aluminum to form ALN via cryo-milling of aluminum with liquid nitrogen can be used to significantly increase yield strength. A-5083 alloy was treated with liquid nitrogen in cryo-milling and was sintered to 99.6% density [12].

2.7. Ball Milling

Ball milling is a process used to blend or mix the materials and it is done in a bottle or drum with balls and material to be process inside it. Ball milling process is mainly used for mineral dressing and ceramic synthesis. It is also an important fabrication process in powder metallurgy.

On laboratory level, two type of ball milling are performed which are low energy ball milling and high energy ball milling. We performed both to blend BN as reinforcement in Al2024 matric and will study the effect of each process on our nanocomposite.

2.7.1 LOW ENERGY BALL MILLING

In low energy ball milling, a bottle containing reinforcement and matrix in powder form is rolled on two rotating rollers. As the bottle is placed horizontally on the rollers, this process also does size reduction of powder particles by the impact of balls when they drop from near top to the bottom of bottle. Ball mills are used extensively in the mechanical alloying process in which they are not only used for size reduction but for cold welding as well, with the purpose of producing alloys from powders.

Different materials are used for milling purpose, including ceramic balls, round rocks like those found in water streams and stainless steel balls. The blending and size reduction is a fucntion of critical speed. Critical speed can be described as the speed after which the balls (added to cause collision with particles and balls) start rotating in the direction in which cylinder is rotating; thus causing no further size reduction.

Some process parameters that should be considered during milling are balls to powder weight ratio, ball size, rpm, and inert atmosphere in bottle so that Aluminum alloy cannot make oxides.

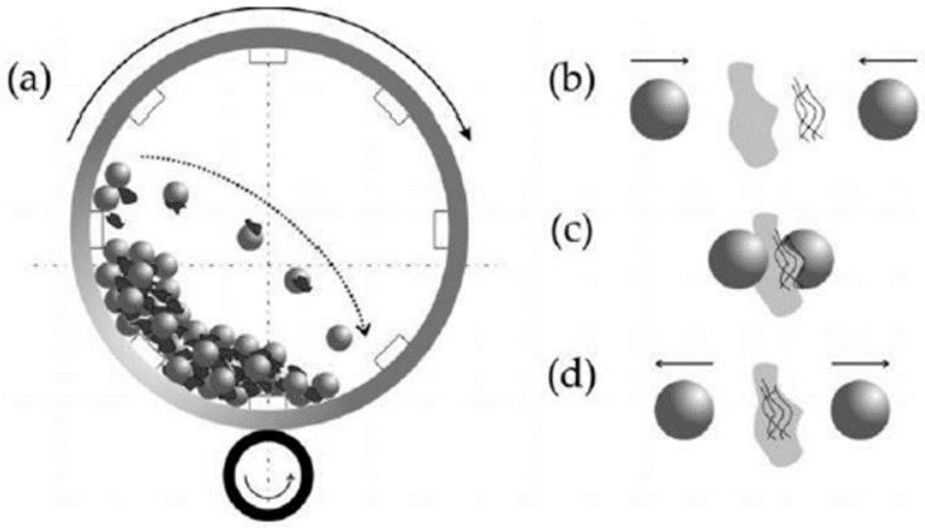

Figure 3 Low energy ball mill

2.7.2 HIGH ENERGY BALL MILLING

High energy ball milling is completely different from low energy ball milling in many aspects. Unlike low energy ball milling, the jars (containing BN and Al2024 alloy in powder form) are placed on a plate which rotates in one direction while the jars spin in counter direction. The advantage of this is that the balls do not gain critical speed and do not start to rotate centrifugally on the walls of jar. High energy ball milling process is emphasized more for blending rather than size reduction.

Varying rotational speeds, ball sizes and materials, gas atmospheres, powder to ball ratios and mixing times can be used as mixing parameters. Process control agents are added to the high energy ball milling process so that cold welding can be avoided. Boric acid can be used as an agent to avoid oxide formation as aluminum and its alloys has high tendency of oxide formation.

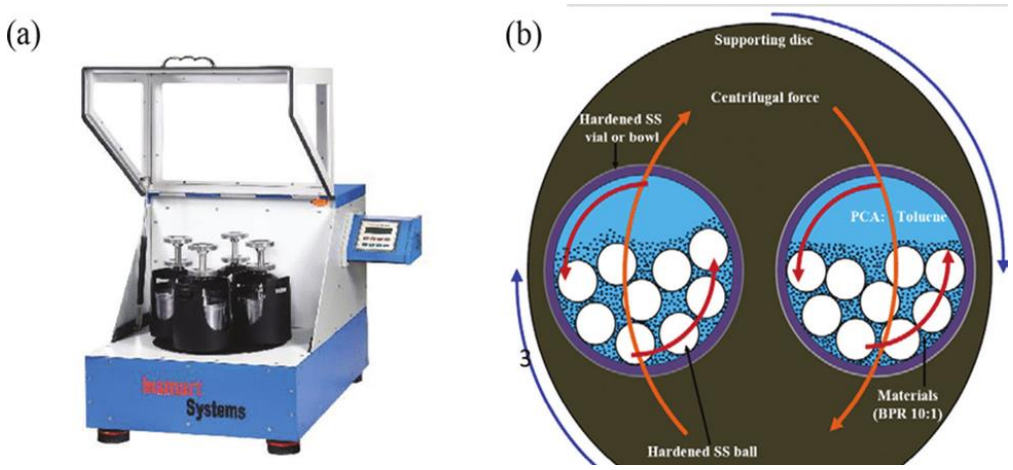

Figure 4 High energy ball mill

CHAPTER 3

EXPERIMENTAL WORK

3.1. Design and fabrication of the die to be used for cold pressing

AutoCAD was used to design the die. Designed Die can produce green samples of 26mm. This size was adequate for testing and fabrication for Compression and Hardness testing.

The die was manufactured on a lathe and subsequently heat treated using D-2 tool steel after finalizing the design. D-2 tool steel was chosen as it provides an effective combination of toughness, hardness, tool performance, corrosion resistance and price.

3.2. Process overview

Although two different routes were used to fabricate composites of the desired composition, the general process employed was to blend the matrix material with the reinforcement, cold press it in a die and sinter it in an inert environment using a tube furnace. The routes differed in the blending step; this is illustrated below:

3.2.1 Experimental Methodology

We used two distinctive routes used to fabricate desired composites

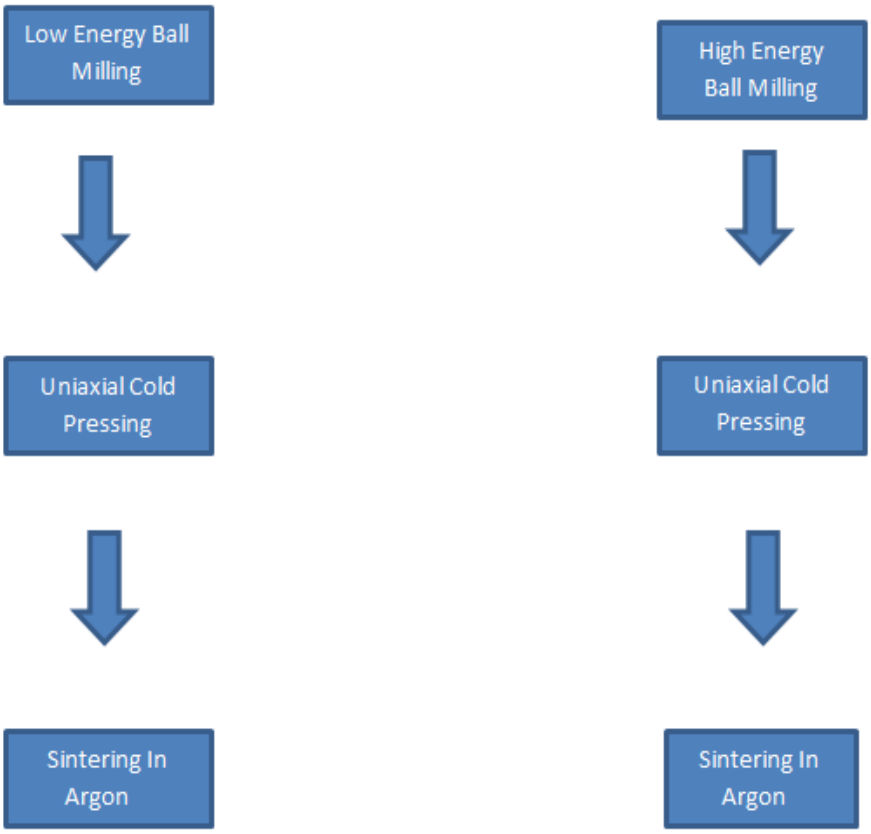

Figure 5 Overall process discribtion

3.2.2 Experimental setup

Aluminum alloy powder exposure to air was avoided where possible throughout the fabrication process due to possible oxidation or contamination. The use of a vacuum glove box in conjunction with Argon gas was employed whenever possible. This allowed processing in an inert environment and prevented oxidation and other undesirable reactions. Cold compaction of milled powder was carried out with a die and hydraulic press.

Next step was sintering of pellets. Green composites were sintered in tube furnace. Tube furnace was connected to vacuum pump and Argon cylinder using pipes. An Erlenmeyer flask filled with viscous synthetic oil

was also used to bubble the outlet argon gas. This was to prevent any backflow of air.

Sintering conditions were optimized for these composites as a function of sintering time and temperature. For this purpose seven different conditions were tested out and then optimal conditions from these were thus chosen for sintering.

3.2.3 Weighing of precursors

Both Aluminum alloy powder and the reinforcement was weighed on a high precision analytical balance to produce the desired the weight fractions in a vacuum glove box. Argon gas connected to the vacuum glove box was used to provide an invert processing environment. Boron nitride as weight percent of 0.1%, 0.2%, 0.4% and 1 % was added in Al matrix.

3.2.4 Blending

Blending was carried out using two distinctive routes explained below.

3.2.4.1 Low energy ball milling

Aluminum alloy powder and different weight composition of Boron nitride was weighed and placed in polypropylene bottles along with steel balls having radius of 5mm in vacuum glove box having Argon environment. Ball to powder ratio were kept in 15:1.

The polypropylene bottles were then placed in ball mill for three different durations for investigation of milling time on both exfoliation and mixing of boron nitride.

Sample powder was milled for 24 hours, 48 hours and 72 hours at 100 RPM.

3.2.4.2 High energy Ball milling

Aluminum alloy powder and different weight composition of Boron nitride was weighed and placed in stainless steel containers specially designed for high impact milling in vacuum glove box having Argon environment. Stainless steel balls of radius 5mm and 10mm were used for milling.

Stearic acid was used as process controlling agent. Stearic acid by weight percentage 0.1 % was added in powder. Ball to powder ratio were kept in 10:1. The container was purged with Argon gas.

Alloy powder was milled for 1 hour, 2 hours and 3 hours at 300 RPM.

3.2.5 Pressing

The mixture powder obtained from both low and high energy milling were placed in a die in vacuum glove box and was then pressed using hydraulic press until compaction was achieved.
300 MPa pressure was applied for 1 minute to ensure even compaction of powder. The green pellets were then removed from die and placed in inert environment to prevent oxidation.

3.2.6 Sinterring

Sintering was integrated into the furnace heating program. First the greens samples are placed in Tube furnace which is connected to vacuum pump for removal of any gas from the tube. Green samples are then heated up to 560 C with heating rate of 10 C/min in inert Argon environment.

The green samples were allowed to soak in this temperature for 180 minutes and then allowed to furnace cool with the gas trapped inside the tube. After furnace cooling samples are then removed from furnace.

3.2.7 Densification

To find the densification and porosity of sintered samples, scanning electron microscope (SEM) and Image-j software was used. SEM images of sintered samples at different time and temperature was analyzed in image-j software to check how much densification is obtained.

3.2.8 Hardness

Vickers hardness test was used for hardness measurement of pellets. Compared to other tests, Vickers test is often easier to perform and calculations are independent of the size of the indenter used for indentation. Besides this, the indenter material is same no matter which

material is being tested and how hard it is. This method is based on an optical measurement system and is performed according to ASTM E92. It Although this test is quite useful for hardness measurements, it requires the sample to be highly polished so that indent is clearly visible and measuring indent is easy. A square base pyramid shaped diamond is used for testing in the Vickers scale. 30 kgf load was used in this method.

3.3 Characterization

The Al-2024/BN obtained after sintering was fed for different characterization techniques. Below is the characterizations which was carried out:

3.3.3 Scanning Electron Microscopy (SEM)

Microstructural analysis was done by Scanning Electron Microscopy. In SEM, an accelerated focused beam of electrons interacts with the surface of the sample. As a result, various interactions between the surface and electrons take place which gives a variety of signals that can be used to obtain the information about surface morphology and topography. The particle morphology along with grain size, grain boundaries, and porosity were analyzed from SEM.

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Optimization of sintering process

4.1.1 SEM micrographs of Al-2024 powder

SEM micrographs of the Aluminum Alloy 2024 powder used throughout are shown below. This is done for verification of the powder size which is indeed < in size and spherical in morphology

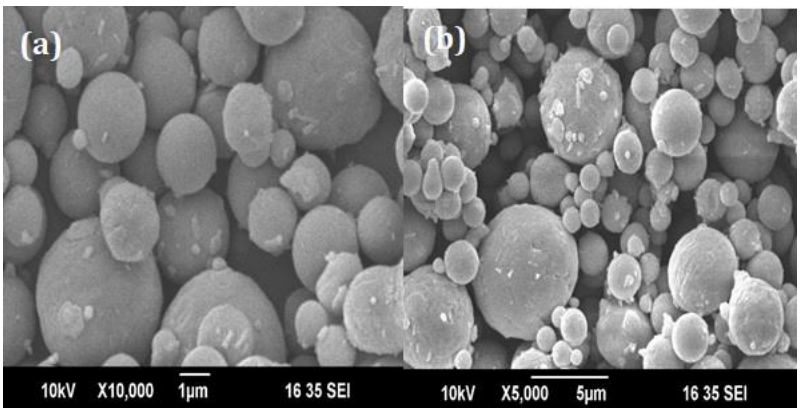

Figure 6 SEM OF AL-2024

4.1.2 SEM micrographs of the Boron Nitride (BN) powder

SEM micrographs of the BN used throughout are shown below. This is done for visual verification.

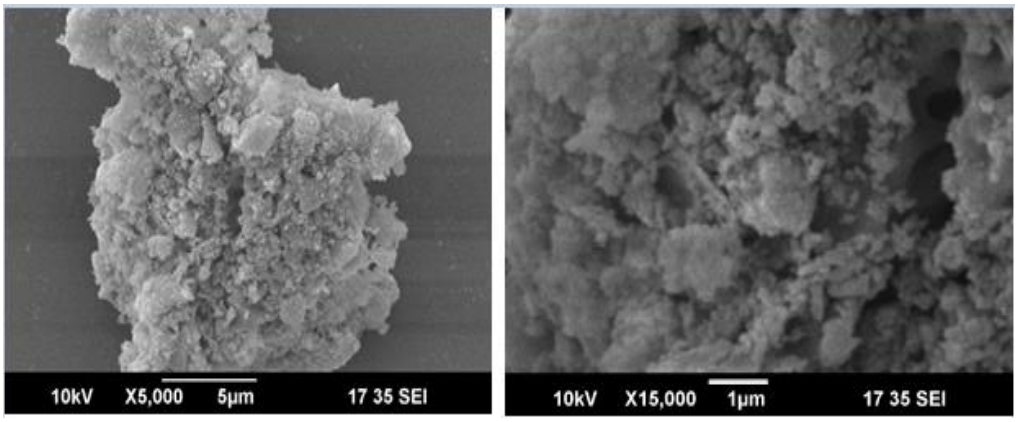

Figure 7 SEM of BN

4.1.3 Optimization of sintering condition

Sintering conditions is optimized for both low energy and high energy ball milling route. For these four sintering temperatures and 2 sintering time is chosen. The sintering temperatures are 500, 530, 550 and 580 and sintering time are 120 minutes and 180 minutes. Sintered pellets are observed via Scanning electron microscope (SEM). Vickers hardness test is also done on these pellets. Densification of these pellets are observed from Image-j software

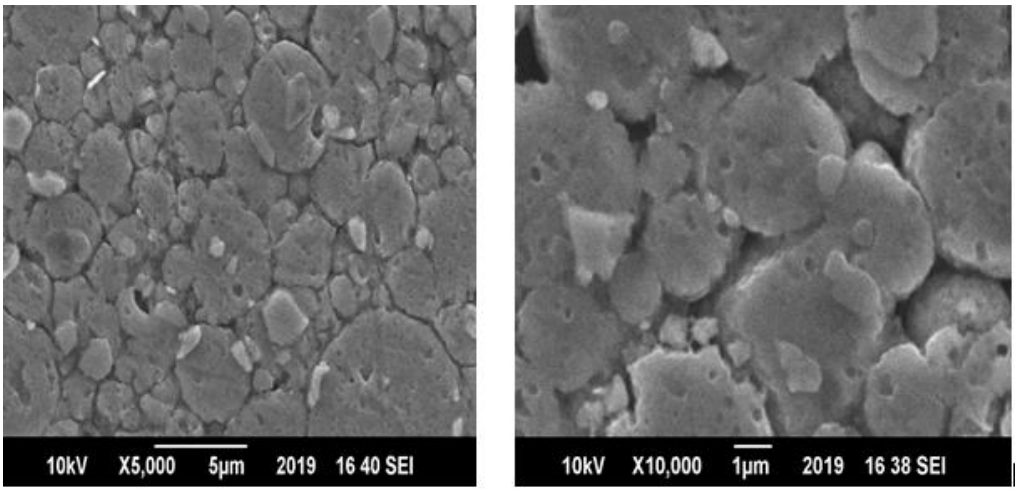

Figure 8 SEM of sintered sample

4.1.4 Densifcation of SEM images by Image J software

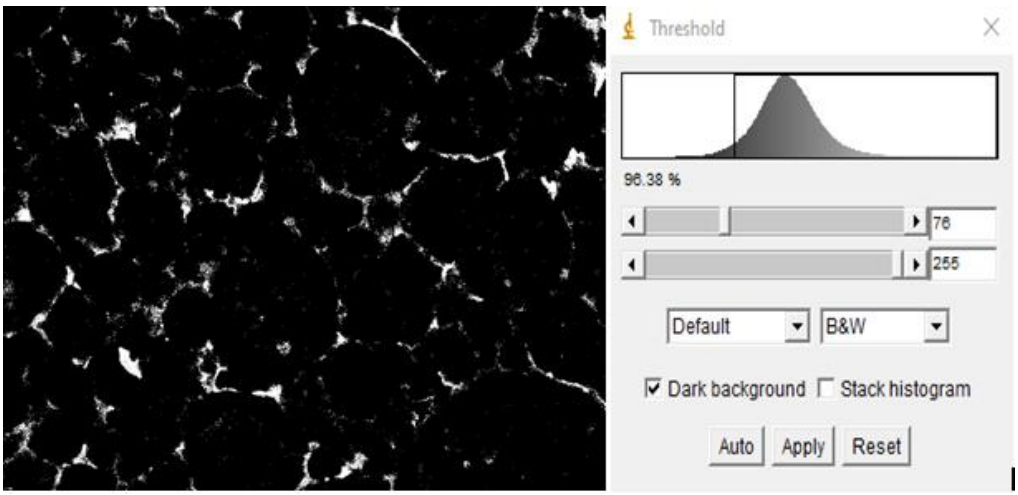

Figure 9 Densification

4.1.5 SEM of sintered pellets at 550℃ for 180 minutes

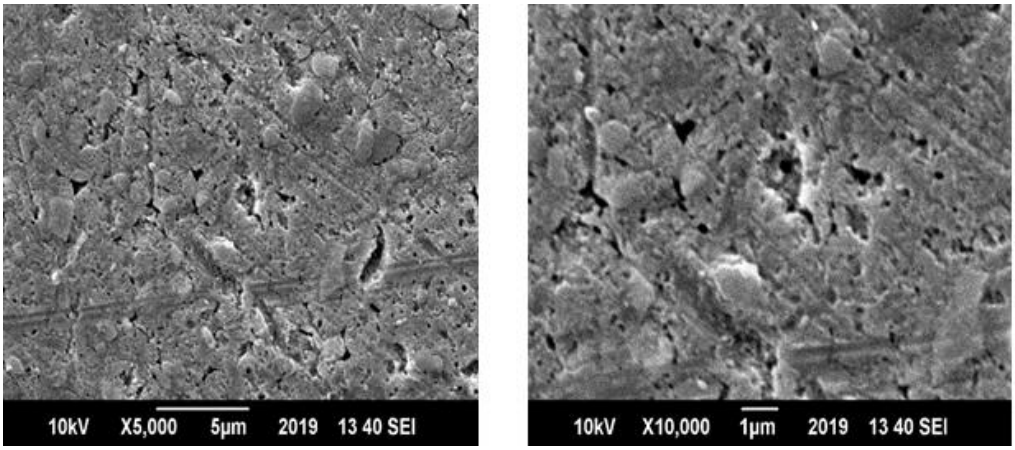

Figure 10 SEM of sintered pallet at 550°C

4.1.6 Densification of SEM images by Image J software

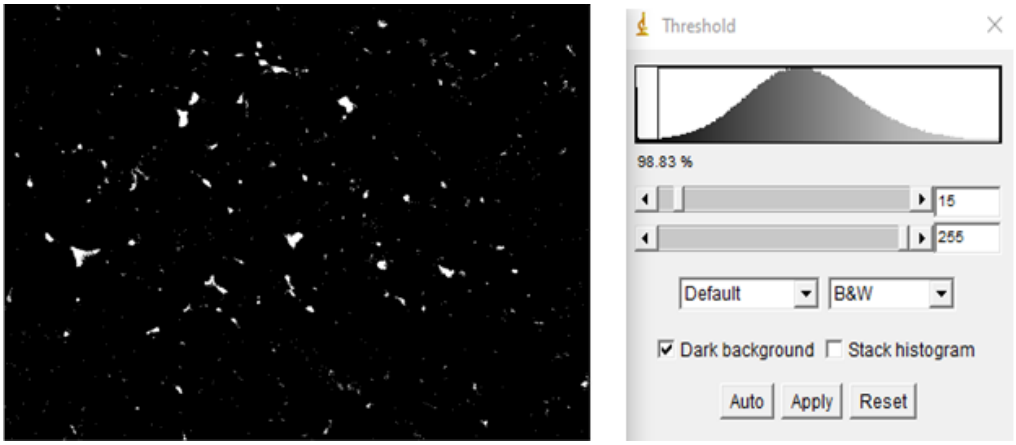

Figure 11 Densification

4.1.7 Hardness variation with changing sintering conditions

When sintering conditions of pure Al-2024 were varied hardness results varied accordingly. The changes in hardness with respect to sintering temperature and sintering time were obserevd and best conditions were choosen for further experimentations on actual composite. It was obsereved that with increase in sintering temperature percentage porosity was decreased and higher hardness values were achieved as shown in graph below. This trend was obsereved till 550°C and at 580°C the hardness value decreased sharply to 200MPa because at this temperature melting was observed in samples.

While chosing sintering time it was obsereved that when time of sintring was increased hardness increaesed for a given temperatre, this is because

grains were given more time to grow and prosity was decreased effectively at higher values of sintering time. At 550°C and highest hardness value was obtained which was 448Mpa for 2hr sinterting time and 496MPa for 3hrs sintering time, respectively.

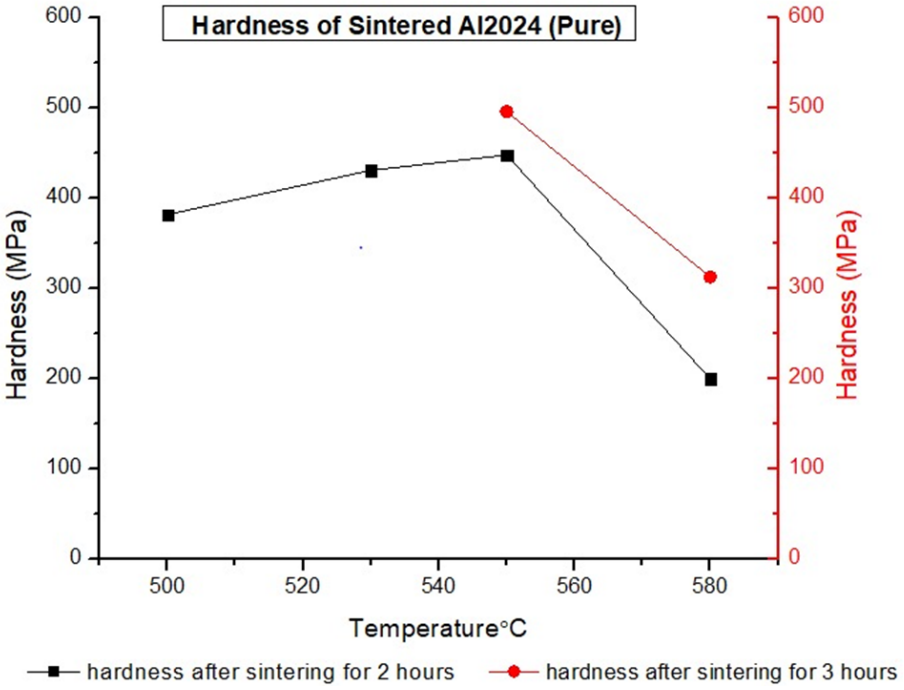

Figure 12 hardness variation with sintering temperature

4.2 Exfoliation

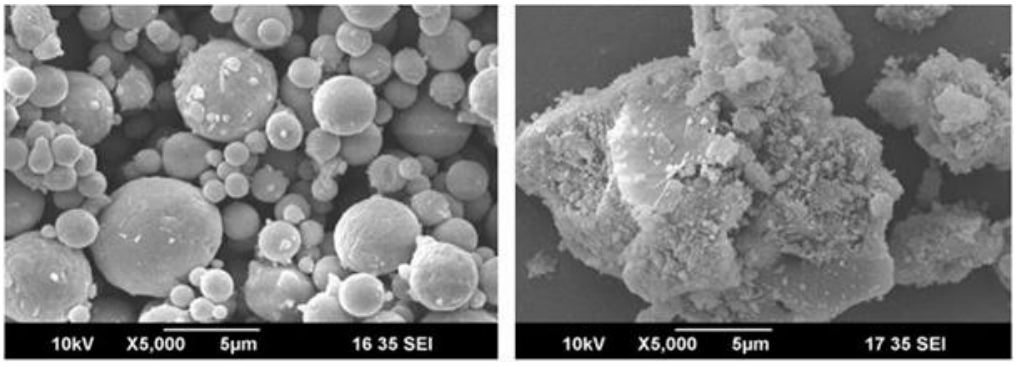

Figure 13 SEM of pure Al and BN

Above are the picture of pure Al 2024 alloy and Boron Nitride (BN) which are not milled yet. Particle size of pure Al 2024 is approximately 5 to 6 micrometers and for BN it is approximately 1 micrometer. As you can see the particle shape of Al 2024 is spherical. BN particles are agglomerated and are in form of flakes.

As we know two types of milling i.e. Low energy Ball Milling and High energy Ball milling was done for mixing of powders and exfoliation of BN. In low energy ball milling time of millings were 24 hours, 48 hours, 72 hours. After milling of 24 hours, 48 hours and 72 hours we see a trend in exfoliation of BN particles in sheet like form i.e. Boron Nitride Nanosheets (BN-NS). We will discuss exfoliation of BN after each time separately. Milling was carried out in inert environment.

As it is a in situ process, we also see blending of Al 2024 alloy particles with BN. This will be shown in upcoming SEM results.

4.2.1 Low energy ball milling exfoliation results

4.2.1.1 Exfoliation of BN after 24 hours

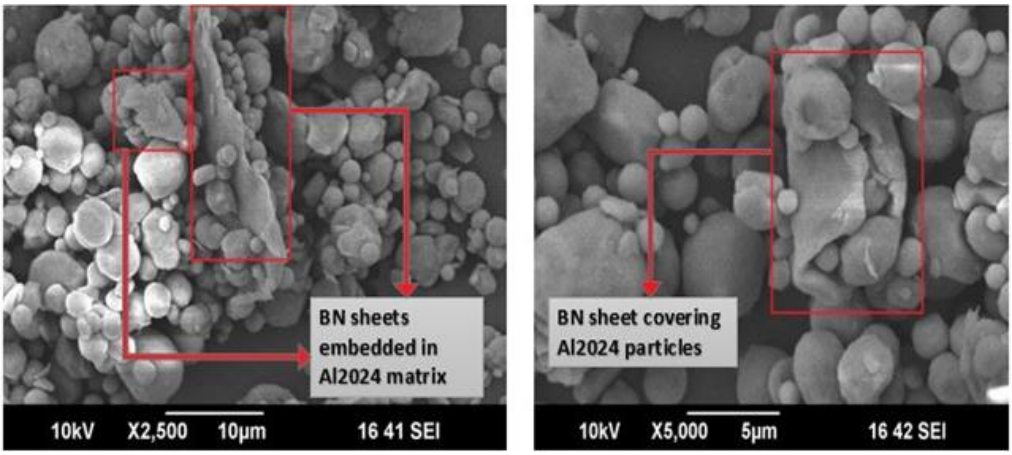

Figure 14 Exfoliation of BN after 24hrs

Above is the SEM result of BN reinforced Al 2024 alloy matrix composite after milling of 24 hours. As we can see from the results BN is exfoliated to sheet like structure which is shown in red box. Al 2024 alloy particles shapes and size are also changed. BN sheets are embedded in Al 2024 particles. In this way they create a bridge like structure in matrix and increase mechanical properties of alloy.

Sometime BN sheets can be seen covering the particles as we can see in second picture. In results we can see Al 2024 alloy particle size is reduced too as compared to pure un-milled Al-2024 alloy. So, mixing and exfoliation can both be seen in the pictures.

4.2.1.2 Exfoliation of BN after 48 hours:

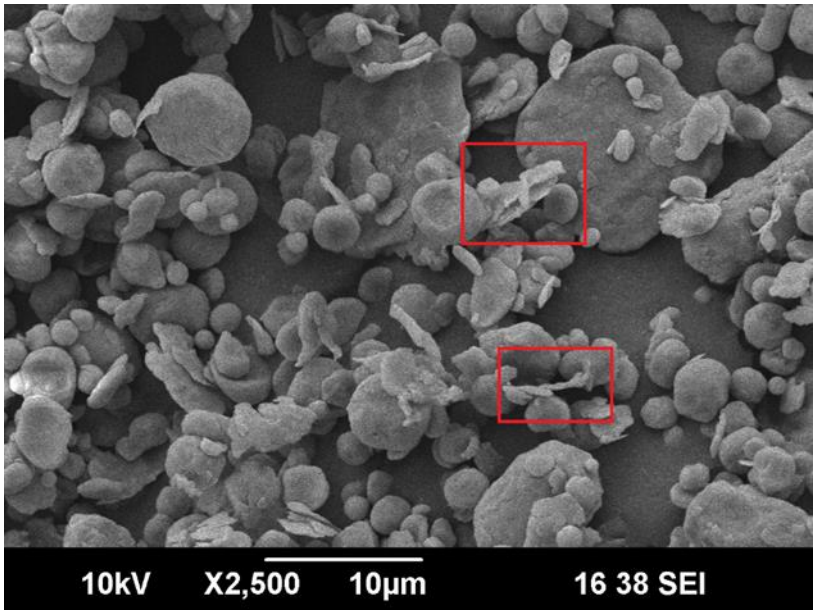

Figure 15 Exfoliation of BN after 48hrs

Above is the result of milling after 48 hours. BN sheets size reduces after increase in milling time. BN Sheet size after 48 hours is less than 24 hours of milling. Al 2024 alloy particles size also reduces. Embedment of BN particles can be seen very clearly now in results.

4.2.1.3 Exfoliation of BN after 72 hours

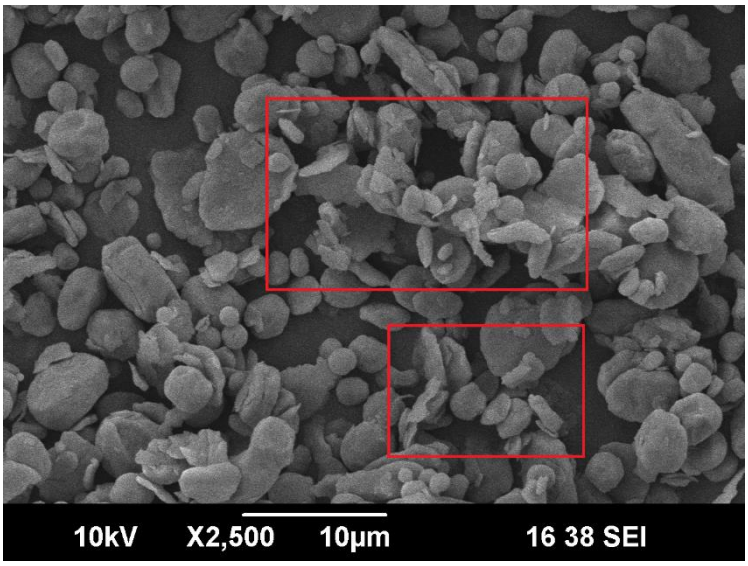

Figure 16 Exfoliation of BN after 72hrs

Above is result of milling after 72 hours. BN particles can be seen in sheet like structure between Al 2024 alloy particles. From SEM results we can see that sheets size is reduced more as compared to 48 and 24 hours of

milling. Al 2024 alloy particles size is also quite reduced as compared to prior milling. BN particles can be seen blended in Al alloy matrix.

From the above results we can see exfoliation and blending increases with increase in milling time.

4.2.2 High Energy Ball Milling Exfoliation results

Moving towards the other processing route, which is high energy ball milling. In case of high energy ball milling Hexagonal Boron Nitride (H-BN) was initially ball milled for 6 hours to reduce its size and after that Al-2024 powder was added and mixing for further 2 hours was done. The reason for dividing a process in two parts was that overheating of Al-2024 powder might result in fire, Thus to reduce risk of fire process was divided in two 2 parts.

4.2.2.1 Exfoliation of pure-BN after 6 hours

Below is the picture of pure BN exfoliated for 6 hours via high energy ball milling route.

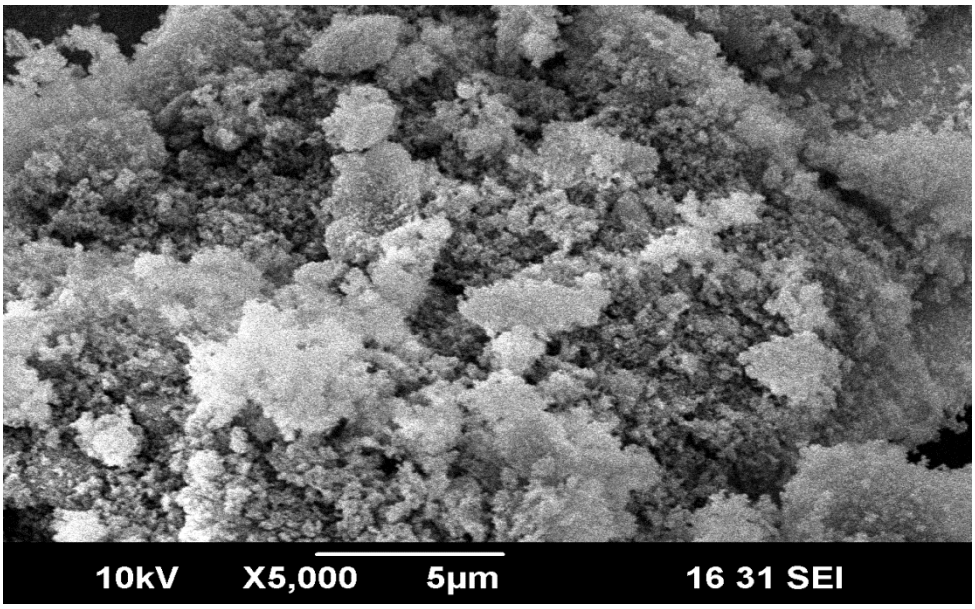

Figure 17 Exfoliation of BN after 6hrs via high energy ball milling

4.2.2.2 Mixing of Al-2024/BN composite for 2 hours

After 6 hours of milling of boron nitride the jars were cooled and AL-2024 powder was added along with 0.1% by weight Stearic acid for avoiding agglomeration and sticking of powder with balls. Below are the SEM

images of Al-2024/BN composite having 0.2%BN weight percent of sample. It can be seen from SEM images that exfoliation is more efficient in case of high energy ball milling compared to low energy ball milling as the size of hexagonal boron nitride (H-BN) nanosheets is reduced more in case of high energy ball milling.

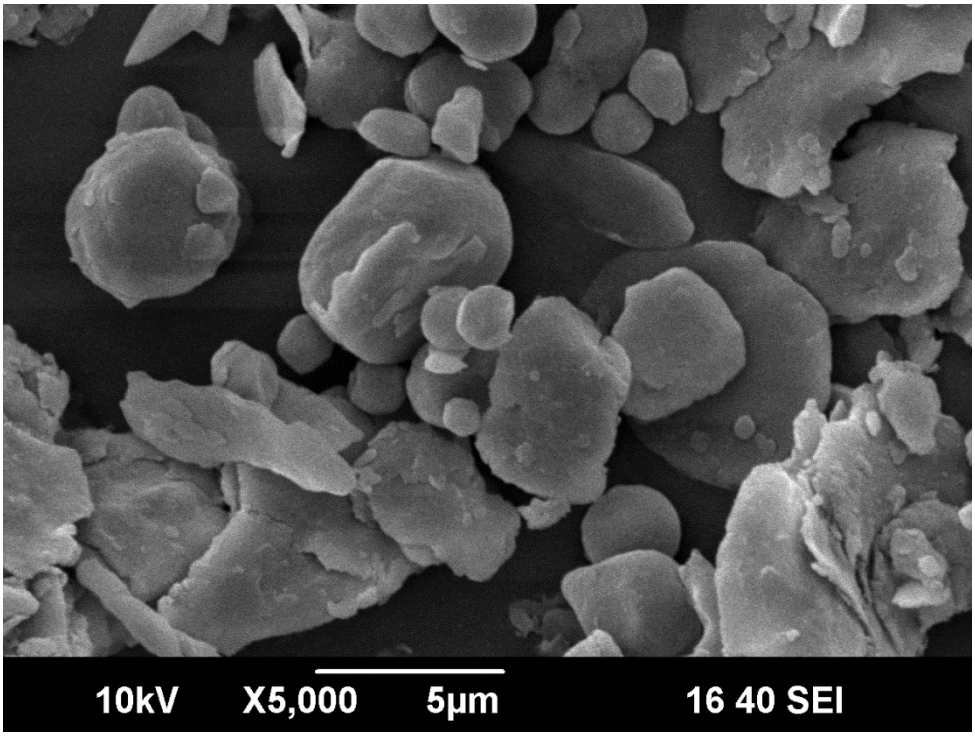

Figure 18 Mixing of Al-2024/BN

4.4 Hardness of Al-2024/BN composites

This section of results is sub-divided in two sections on basis of type of ball milling used. The former section explains results obtained from low energy ball milling while the later deals with results obtained from high energy ball milling. In both cases to check hardness Vickers hardness test was implied with load of 30kgf and dwell time was kept 12 seconds respectively. The standard followed is ASTM E92**.**

4.4.1 Hardness for Low energy ball milling

Below are the results obtained from Vickers hardness test, milling times ranging from 24hrs, 48hrs and 72hrs were chosen and after optimization 72hrs milling time was opted for further testing. The graph shows a trend of change in hardness with respect to change in percentage of boron

nitride in Al-2024 powder keeping the milling time constant i.e. 72hrs. A maximum hardness obtained was 814MPa when 0.2% BN weight fraction of sample was added in AL-2024 matric. On further increase in percentage of boron nitride (BN) shows that exfoliation was not affective via low energy ball milling route and hardness tends to decrease with increase in percentage of boron nitride (BN). The reason for this decrease is agglomeration of nano-sheets of boron nitride. Thus hardness values achieved by adding 1%BN weight fraction of sample hardness value was 627MPa which is far less than the value achieved when only 0.2%BN weight fraction of sample was added.

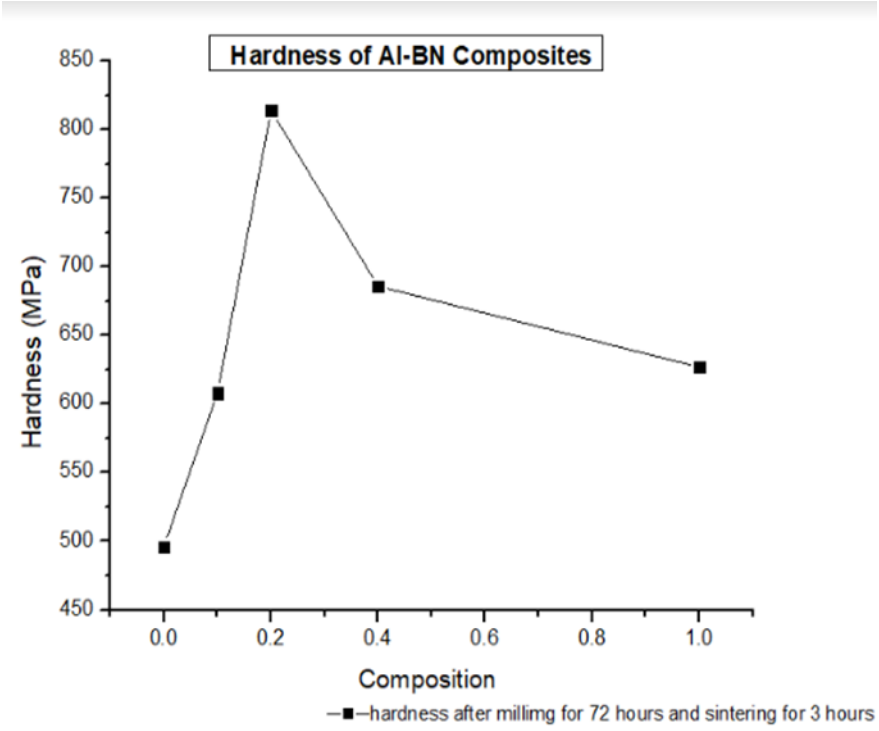

Figure 19 Hardness via low energy ball mill

4.4.2 Hardness for high energy ball milling:

When Al-2024/BN composite was ball milled via high energy ball milling, it was observed that hardness value increased for a given composition of BN. The hardness test was applied on 0.2%BN weight fraction of sample and 0.4%BN weight fraction of sample. The reason of higher hardness values for high energy milling is that may be exfoliation of boron nitride nano-sheets was more efficient in case of high energy milling compared to

low energy milling. The hardness value increased with increase in milling time as shown in graph below:

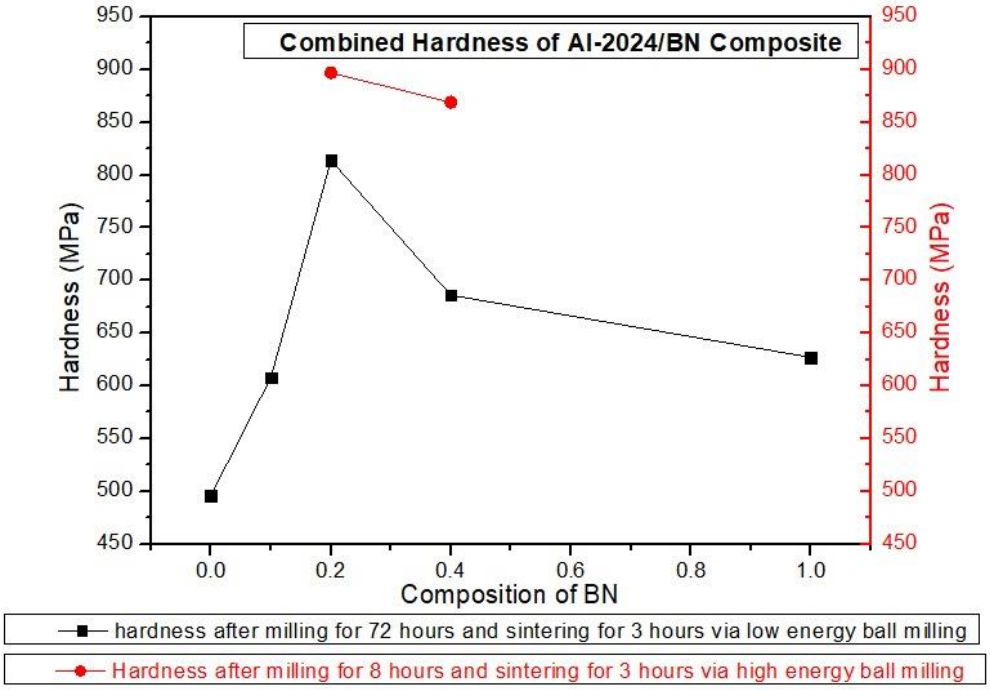

Figure 20 Combined hardness

CONCLUSIONS

• Boron Nitride particles exfoliate into Nanosheets in both High and Low energy ball milling with increasing time of milling

• Hardness of BN reinforced Al metal matrix composite increases with increasing time of milling and weight composition of reinforcement upto 0.2% Boron Nitride

• Densification and hardness increase when we are optimizing sintering temperature and time. The temperature and time at which we got highest hardness and densification value is the optimum temperature and time

REFERENCES

[1] J. W. White, ‘‘Handling aluminum powders: safety issues.’’ JWW/ALPOSAF/12/98,
The Aluminum Powder Company Limited, Dec. 1998.

[2] S. S. Grossel, ‘‘Fire and explosion hazards in powder handling and processing.’’ Handbook
of Powder Science, pp. 854^868.

[3] ASM Handbook, Volume 2: Properties and Selection: Nonferrous Alloys and Special-Purpose Materials ASM Handbook Committee, p 200-215

[4] ASM Handbook, Volume 7: Powder Metal Technologies and Applications P.W. Lee, Y. Trudel, R. Iacocca, R.M. German, B.L. Ferguson, W.B. Eisen, K. Moyer,D. Madan, and H. Sanderow, editors, p 834-839

[5] Pieczonka, T. & Schubert, Thomas & Baunack, Stefan & Kieback, Bernd. (2005). Sintering Behaviour of Aluminium in Different Atmospheres.

[6] Borkar T, Hwang J, Hwang JY, et al. Strength versus ductility in carbon nanotube reinforced nickel matrix nanocomposites. J Mater Res 2014; 29: 761–769.

[7] Schaffer, G.B., Hall, B.J. The influence of the atmosphere on the sintering of aluminum. Metall and Mat Trans A 33, 3279–3284 (2002).

[8] A. R. Begg, ‘‘Development, Manufacture and Properties of PM Aluminum-Based Metal
Matrix Composites,’’ International Conference on PMAerospaceMaterials 1991.

[9] Grishin, V.. (1964). Brazing SAP-2 Sintered Aluminum Powder. 9.

[10] V. G. Gopienko, Val. G. Gopienko, and Y. G. Olesov, ‘‘Aluminum Alloy Powders for
the Production of Sintered Materials (Review),’’ All-Union Aluminum and Magnesium
Institute, Translated from Poroshkovaya Metallurgiya, 1985, 4(268), pp. 24^28.

[11] A. R. Begg, ‘‘Metal-Matrix Composites by Powder Metallurgy,’’ Powder Metallurgy,
1993, 36(2), pp. 107^110.
[12] Aluminum Association, ‘‘Guidelines for handling aluminum cnes generated during various
aluminum fabricating operations.’’ Brochure F-1.
[13] S. S. Grossel, ‘‘Fire and explosion hazards in powder handling and processing.’’ Handbook
of Powder Science, pp. 854^868.
[14] Aluminum Association, ‘‘Recommendations for storage and handling of aluminum
powder and paste.’’ Brochure TR-2.
[15] A. R. Begg, ‘‘Metal-Matrix Composites by Powder Metallurgy,’’ Powder Metallurgy,
1993, 36(2),
[16] Powder Metallurgy Design Manual, 3rd Edn, 1998, MPIF, p. 24.
[17] ‘‘Sintering of Aluminum and Aluminum Alloys,’’ ASM Handbook, Vol. 7, 1998, Powder Metal Technologies and Applications, ASM International, pp. 381^385.
[18] W. H. Hunt Jr., ‘‘Cost-Effective High Performance P/MAluminum Matrix Composites
for Aerospace Applications’’. International Conference on PM Aerospace Materials,
1991, Paper 32.
[19] Xue ZW, Wang LD, Zhao PT, et al. Microstructures and tensile behavior of carbon nanotubes reinforced Cu matrix composites with molecular-level dispersion. Mater & Des 2012; 34: 298–301.
[20] Pham VT, Bui HT, Tran BT, et al. The effect of sintering temperature on the mechanical properties of a Cu/CNT nanocomposite prepared via a powder metallurgy method. Adv Nat Sci Nanosci Nanotechnol 2011; 2: 015006.