DRY AND WET GRINDING BEHAVIOUR OF Fe AND SiO_2 IN SPECIALLY DESIGNED DUAL DRIVE PLANETARY BALL MILL

A thesis submitted in partial fulfillment
Of the requirement for the degree of
Bachelor of Technology
In
Metallurgical and Materials Engineering
By

SUBHAM GARG (110MM0522)

Under the guidance of

DR. DEBASIS CHAIRA

METALLURGICAL AND MATERIALS ENGINEERING
NATIONAL INSTITUTE OF TECHNOLOGY
ROURKELA (2014-2015)

NATIONAL INSTITUTE OF TECHNOLOGY ROURKELA

CERTIFICATE

This is to certify that the thesis entitled "DRY AND WET GRINDING BEHAVIOUR OF Fe AND SIO_2 IN SPECIALLY DESIGNED DUAL DRIVE PLANETARY BALL MILL" being submitted by SUBHAM GARG (110MM0522) for the partial fulfillment of the requirements of Bachelor of Technology degree in Metallurgical and Materials Engineering is a bona fide thesis done by him under my supervision during the academic year 2014-2015, in the Department of Metallurgical and Materials Engineering, National Institute of Technology Rourkela, India.

The results presented in this thesis have not been submitted elsewhere for the award of any other degree or diploma.

DATE: 19/11/2014

(DR. DEBASIS CHAIRA)
METALLURGICAL AND MATERIALS ENGINEERING
NATIONAL INSTITUTE OF TECHNOLOGY
ROURKELA, ODISHA-769008

ACKNOWLEDGMENT

I would like to express my sincere gratitude to my guide Dr. Debasis Chaira, Metallurgical and Materials Engineering, NIT Rourkela, for giving me the opportunity to work with him and also providing excellent guidance and continuous assistance throughout the project work. His constant criticism, advice, assertions, appreciation were very vital and irrevocable, giving me that boost without which it wouldn't have been possible for me to finish my project. There is not a single day i can remember during my work days which went by without his scrutiny and guidance. I have received endless support and guidance from him, right from the development of ideas, deciding the experiments and methodology of work and this presentation. I am thankful to him for his encouragement throughout the project. I wish to express my deep sense of gratitude to Dr. S. C. Mishra, HOD, Metallurgical and Materials Engineering, NIT Rourkela for giving me an opportunity to work on this project. I would be highly obliged to extend my thanks to Ph.D. scholar Mr. Shashanka R and the M. Tech student Mr. George Lenin T M for their immense support and help rendered while carrying out my experiments, without which the completion of this project would have been at stake. I would also like to thank all the staff members of MME Dept., NITR and everyone who in some way or the other has provided me valuable guidance, suggestion and help for this project.

DATE: 19/11/2014

SUBHAM GARG (110MM0522)

METALLURGICAL AND MATERIALS ENGINEERING

NATIONAL INSTITUTE OF TECHNOLOGY

ROURKELA, ODISHA-769008

ABSTRACT

In the project, an attempt has been made to observe the dry and wet grinding behavior of ductile material - iron (Fe) and brittle material - sand (SiO_2) in dual drive planetary ball mill for 0h, 15min, 30min, 1h, 1/2h, and 2h through mechanical alloying. The phase transformation occurring in the materials during milling were studied using X-RAY diffraction. Particle size analysis was carried out to study the size reduction of particles as a function of milling time and it has been found that at the initial milling period size reduction is very low in case of iron (Fe) due to its ductile nature, and size reduction is very fast in case of sand (SiO_2) due to its brittle nature. It was also found that less fine particles are formed during dry milling than wet milling due to formation of agglomerates.

KEYWORDS: Mechanical Alloying, Dual Drive Planetary Mill, X-RAY Diffraction, Particle size analysis.

LIST OF FIGURES

CONTENT

1. INTRODUCTION

Mechanical alloying was used first in the early 1970s for the production of oxide-dispersion strengthened (ODS) super alloys. It is now recognized as a versatile technique for the production of a broad range of powders, from amorphous or Nano crystalline to ODS or intermetallic among others. Planetary ball milling is carried out for fabrication of engineering materials via a mechanical alloying process. Mechanical alloying (MA) is a high energy ball milling process by which constituent powders are repeatedly deformed, fractured and welded by grinding media to form a homogeneous alloyed microstructure or uniformly dispersed particulates in a matrix, The main objectives of the milling process are to reduce the particle sizes (breaking down the material), mixing, blending and particle shaping. The process requires at least one fairly ductile metal (e.g. Aluminium) to act as a host or binder.

Non-equilibrium processing of materials has attracted the attention of a number of scientists and engineers due to the possibility of producing better and improved materials than is possible by conventional methods [1]. Mechanical alloying (MA) is such processing method. MA started as an industrial necessity in 1966 to produce oxide dispersion strengthened (ODS) nickel- and iron-based super alloys for applications in the aerospace industry and it is only recently that the science of this “apparently” simple processing technology has begun to be investigated. The technique of MA was used for industrial applications from the beginning and the basic understanding and mechanism of the process is beginning to be understood only now. There have been several reviews and conference proceedings on this technique too [2] and [3], but the present status of MA has been most recently reviewed by Suryanarayana [4].

Powder particles during mechanical alloying are subjected to high energy collision, which causes them to be cold welded together and fractured. Cold welding and fracturing enable the powder particles to be always in contact with each other with atomically clean surfaces and with minimized diffusion distance. Micro structurally, the MA process can be divided into three stages: at the initial stage, the powder particles are cold-welded together to form a laminated structure. The chemical composition of the composite particles varies significantly within the particles and from particle to particle. At the second stage, the laminated structure is further refined as fracture takes place. The thickness of the lamellae is decreased. Although dissolution may have taken place, the chemical composition of the powders is still not homogeneous: a very fine crystalline size can be observed. At the final stage, the lamellae become finer and eventually disappear. A homogenous chemical composition is achieved for all particles, resulting in a new alloy with a composition corresponding to that of the initial powder mixture.

This is a solid state powder processing technique which is generally performed in a high-energy ball mill to produce composite powders containing homogeneously distributed alloying element in the matrix. This process avoids many problems associated with conventional melting and solidification [5] and [6]. During ball milling, powder particles are trapped between the rapidly colliding balls where soft metal powder particles are cold-welded and the alloying elements are trapped along the weld interface of soft composite particles [7]. The cold welding results in a built-up of a large powder particle, especially in the early stage of milling, whereas fracturing breaks down the composite powder particles. Further milling leads to a balance, between the cold welding and fracturing, so that the overall average particle size of the milled powder remains constant. This process increases the internal energy of particles and on further sintering second phase particles readily form within the matrix [7].

1.1 Historical Perspectives

Mechanical alloying (MA) is a powder processing technique that allows production of homogeneous materials starting from blended elemental powder mixtures. John Benjamin and his colleagues at the Paul D. Merica Research Laboratory of the International Nickel Company (INCO) developed the process around 1966. The technique was the result of a long search to produce a nickel-base super alloy, for gas turbine applications, that was expected to combine the high temperature strength of oxide dispersion and the intermediate-temperature strength of gamma-prime precipitate. The required corrosion and oxidation resistance was also included in the alloy by suitable alloying additions [8].

2. LITERATURE SURVEY

2.1 Stages of Mechanical Alloying

The different stages of mechanical alloying are:

- Particle flattening

This is the first stage of milling and initially the particles get flattened and become flake like.

- Welding predominance

During the second stage the flattened particles weld to form lamellar or layered composite particles.

- Equiaxed particle formation

After this, the lamellar particles cease to be flake like and become thicker and rounded. The shape change is caused by the work hardening of the powders.

- Random welding orientation

Welding of particles again starts as the fragments from the equiaxed particles start to weld in different orientations and the lamellar structure starts degrading.

- Steady state processing

Ultimately, the structure of the material gets gradually refined as fragments are taken from the particles that later weld with other fragments in different orientations [5].

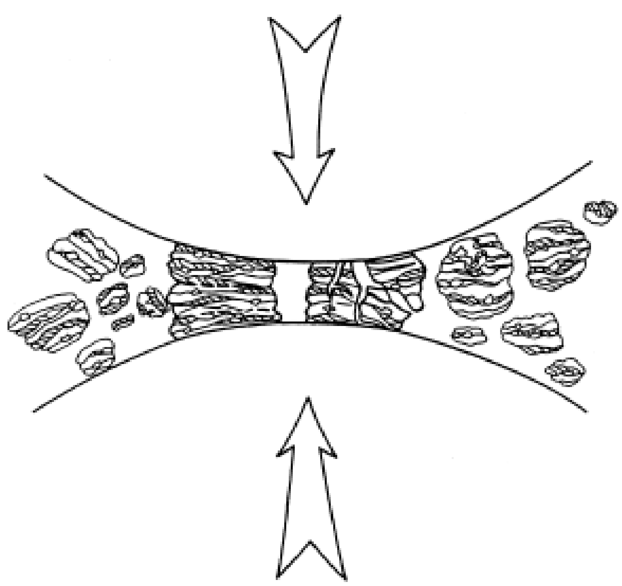


Fig. 1: Ball-powder-ball collision of powder mixture during reaction milling process [5]

2.2 Attributes of Mechanical Alloying

- Production of dispersion of second phase (usually oxide) particles
- Extension of solid solubility limits
- Refinement of grain sizes down to the nanometer range
- Synthesis of novel crystalline and quasicrystalline phases
- Development of amorphous (glassy) phases
- Disordering of ordered intermetallic
- Possibility of alloying of difficult to alloy elements

2.3 Process of Mechanical Alloying

The most important components in MA are as follows:

- Raw Materials
- Ball Mill
- Types of Mill
- Process variables

The process of MA starts with mixing of the powders in the required proportion and loading the powder mix into the mill along with the grinding medium (generally steel balls). The mix obtained is then milled for the desired length of time until a steady state is reached. Steady state is the state when the composition of every powder particle is the same as the proportion of the elements in the starting powder mix. The milled powder is then consolidated into a bulk shape and heat treated to obtain the desired microstructure and properties [8].

2.3.1 Raw Materials:

Generally raw materials with particle sizes in the range of 1-2000nm are used for MA. The powder particle size decreases exponentially with time and reaches a small value of a few microns only after a few minutes of milling. So, powder particle size is not very critical, except that it should be smaller than the grinding ball size.

The raw powders fall into the broad categories of pure metals, master alloys, pre-alloyed powders and refractory compounds. Dispersion strengthened materials usually contain additions of carbides, nitrides, and oxides. Oxides are the most common and these alloys are known as oxide-dispersion strengthened (ODS) materials.

Earlier, the powder charge always consisted of at least 15 vol. % of a ductile compressible deformable metal powder to act as a host or a binder. However, in recent years, mixtures of fully brittle materials have been milled successfully resulting in alloy formation. Thus, the requirement of having a ductile metal powder during milling is no longer necessary. Hence, ductile-ductile, ductile-brittle and brittle-brittle powder mixtures are milled to produce novel alloys. Sometimes, metal powders are milled with a liquid medium and this is referred to as wet grinding. If no liquid is involved then it is referred to as dry grinding [3]. Another kind of wet milling is Cryomilling, where the liquid used is at cryogenic temperature. In case of wet grinding the solvent molecules are adsorbed on the newly formed surfaces of the particles and lower their surface energy. So, wet grinding is better method than dry grinding to obtain finer-ground products. The less-agglomerated condition of the powder particles in the wet condition is also a useful factor. Moreover, it has been reported that the rate of amorphization is faster during wet grinding than during dry grinding. But, the disadvantage of the wet grinding is that an increased contamination of the powder occurs due to the sticking. Thus, most of the MA/MM operations are generally carried out in dry conditions [2].

2.3.2 Ball Mill:

A ball mill, a type of grinder, is a cylindrical device used in grinding (or mixing) materials like ores, chemicals, ceramic raw materials and paints. Ball mills rotate around a horizontal axis, partially filled with the material to be ground plus the grinding medium. Different materials are used as media, including ceramic balls, flint pebbles and stainless steel balls. An internal cascading effect reduces the material to a fine powder. Industrial ball mills can operate continuously, fed at one end and discharged at the other end. Large to medium-sized ball mills are mechanically rotated on their axis, but small ones normally consist of a cylindrical capped container that sits on two drive shafts (pulleys and belts are used to transmit rotary motion). A rock tumbler functions on the same principle. Ball mills are also used in pyrotechnics and the manufacture of black powder, but cannot be used in the preparation of some pyrotechnic mixtures such as flash powder because of their sensitivity to impact. High-quality ball mills are potentially expensive and can grind mixture particles to as small as 5 nm, enormously increasing surface area and reaction rates. The grinding works on the principle of critical speed [2]. The critical speed can be understood as that speed after which the steel balls (which are responsible for the grinding of particles) start rotating along the direction of the cylindrical device; thus causing no further grinding. Ball mills are used extensively in the mechanical alloying process in which they are not only used for grinding but for cold welding as well, with the purpose of producing alloys from powders.

The ball mill is a key piece of equipment for grinding crushed materials, and it is widely used in production lines for powders such as cement, silicates, refractory material, fertilizer, glass ceramics, etc. as well as for ore dressing of both ferrous non-ferrous metals. The ball mill can grind various ores and other materials either wet or dry. There are two kinds of ball mill, grate type and over fall type due to different ways of discharging material. There are many types of grinding media suitable for use in a ball mill, each material having its own specific properties and advantages. Key properties of grinding media are size, density, hardness, and composition [8].

- Size: The smaller the media particles, the smaller the particle size of the final product. At the same time, the grinding media particles should be substantially larger than the largest pieces of material to be ground.
- Density: The media should be denser than the material being ground. It becomes a problem if the grinding media floats on top of the material to be ground.
- Hardness: The grinding media needs to be durable enough to grind the material, but where possible should not be so tough that it also wears down the tumbler at a fast pace.
- Composition: Various grinding applications have special requirements. Some of these requirements are based on the fact that some of the grinding media will

be in the finished product. Others are based in how the media will react with the material being ground.

- Where the color of the finished product is important, the color and material of the grinding media must be considered.
- Where low contamination is important, the grinding media may be selected for ease of separation from the finished product (i.e.: steel dust produced from stainless steel media can be magnetically separated from non-ferrous products). An alternative to separation is to use media of the same material as the product being ground.
- Flammable products have a tendency to become explosive in powder form. Steel media may spark, becoming an ignition source for these products. Either wet-grinding, or non-sparking media such as ceramic or lead must be selected.
- Some media, such as iron, may react with corrosive materials. For this reason, stainless steel, ceramic, and flint grinding media may each be used when corrosive substances are present during grinding.

The grinding chamber can also be filled with an inert shield gas that does not react with the material being ground, to prevent oxidation or explosive reactions that could occur with ambient air inside the mill [8].

2.3.3 Types of Mills:

Different types of high-energy milling equipment are used to produce mechanically alloyed powders. They differ in their capacity, efficiency of milling and additional arrangements for cooling, heating etc.

Some of the mills are:

- SPEX shaker mills
- Planetary ball mills
- Attrition mills
- Commercial mills

SPEX Shaker Mill:

Shaker mills such as SPEX mills, which mill about 10–20 g of the powder at a time, are most commonly used for laboratory investigations and for alloy screening purposes. The common variety of the mill has one vial, containing the sample and grinding balls, secured in the clamp and swung energetically back and forth several thousand times a minute. The back-and-forth shaking motion is combined with lateral movements of the ends of the vial, so that the vial appears to be describing a figure 8 or infinity sign as it moves [4]. With each swing of the vial the balls impact against the sample and the end of the vial, both milling and mixing the sample. Because of the amplitude (about 5 cm) and speed (about 1200 rpm) of the clamp motion, the ball velocities are high (on the order of 5 m/s) and consequently the force of the ball's impact is unusually great. Therefore, these mills can be considered as high-energy variety [8].

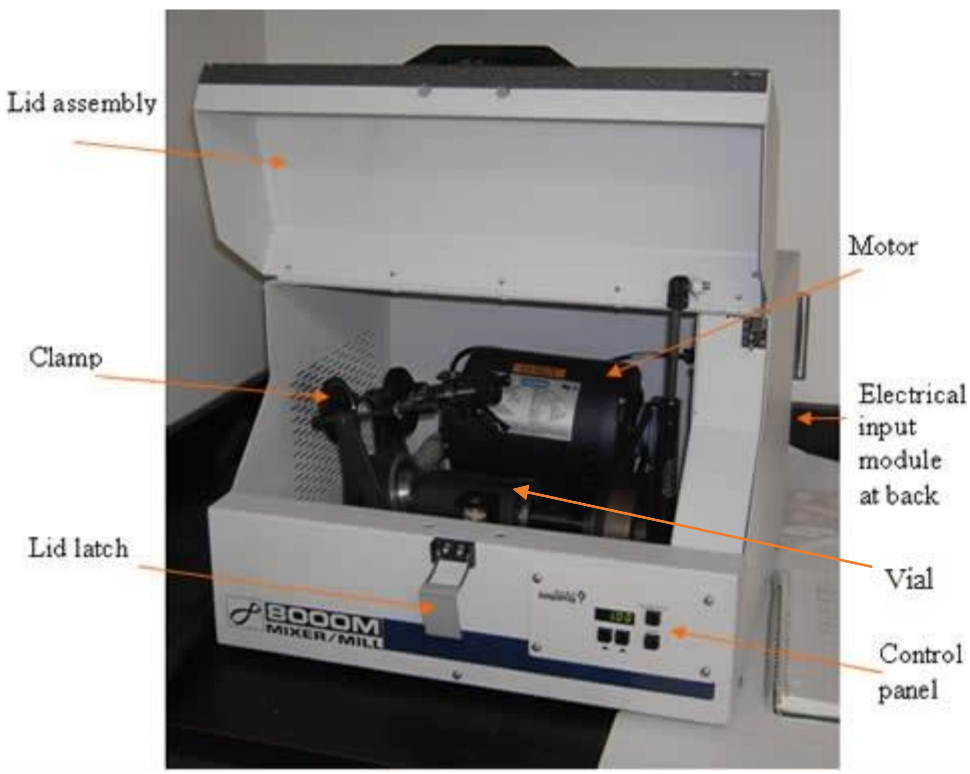
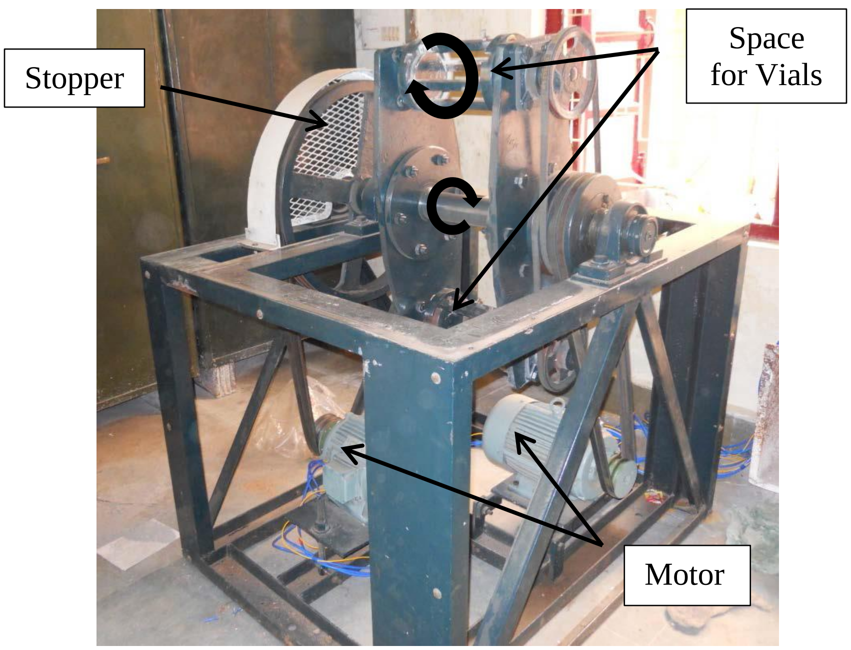


Fig. 2: SPEX Shaker Mill [8]

Planetary Ball Mill:

Planetary ball mill is one in which a few hundred grams of the powder can be milled at a time. The planetary ball mill owes its name to the planet-like movement of its vials. These are arranged on a rotating support disk and a special drive mechanism causes them to rotate around their own axes [8]. The centrifugal force produced by the vials rotating around their own axes and that produced by the rotating support disk both act on the vial consents, consisting of material to be ground and the grinding balls. Since the vials and the supporting disk rotate in opposite directions, the centrifugal forces alternately act in like and opposite directions. This causes the grinding balls to run down the inside wall of the vial — the friction effect, followed by the material being ground and grinding balls lifting off and traveling freely through the inner chamber of the vial and colliding against the opposing inside wall.



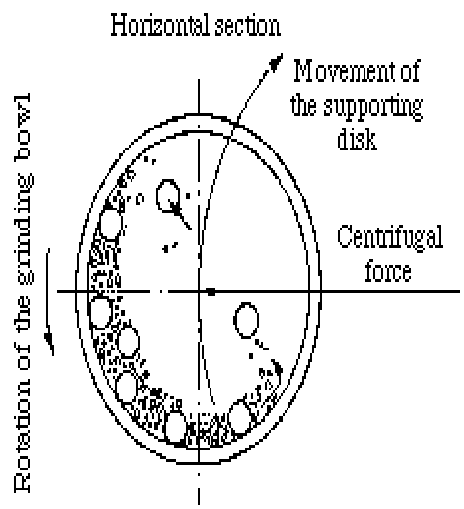


Fig. 3: Planetary Ball Mill

Attrition Mill:

A conventional ball mill consists of a rotating horizontal drum half-filled with small steel balls. As the drum rotates the balls drop on the metal powder that is being ground; the rate of grinding increases with the speed of rotation. At high speeds, however, the centrifugal force acting on the steel balls exceeds the force of gravity, and the balls are pinned to the wall of the drum. At this point the grinding action stops. An attritor (a ball mill capable of generating higher energies) consists of a vertical drum with a series of impellers inside it [1]. Set progressively at right angles to each other, the impellers energize the ball charge, causing powder size reduction because of impact between balls, between balls and container wall, and between balls, agitator shaft, and impellers. Some size reduction appears to take place by inter particle collisions and by ball sliding. A powerful motor rotates the impellers, which in turn agitate the steel balls in the drum.

Attritors are the mills in which large quantities of powder (from about 0.5 to 40 kg) can be milled at a time. Commercial attritors are available from Union Process, Akron, OH. The velocity of the grinding medium is much lower (about 0.5 m/s) than in Fritsch or SPEX mills and consequently the energy of the attritors is low. Attritors of different sizes and capacities are available. The grinding tanks or containers are available either in stainless steel or stainless steel coated inside with alumina, silicon carbide, silicon nitride, zirconia, rubber, and polyurethane. A variety of grinding media also is available — glass, silicon carbide, silicon nitride, alumina, stainless steel, carbon steel, chrome steel, and tungsten carbide [8].

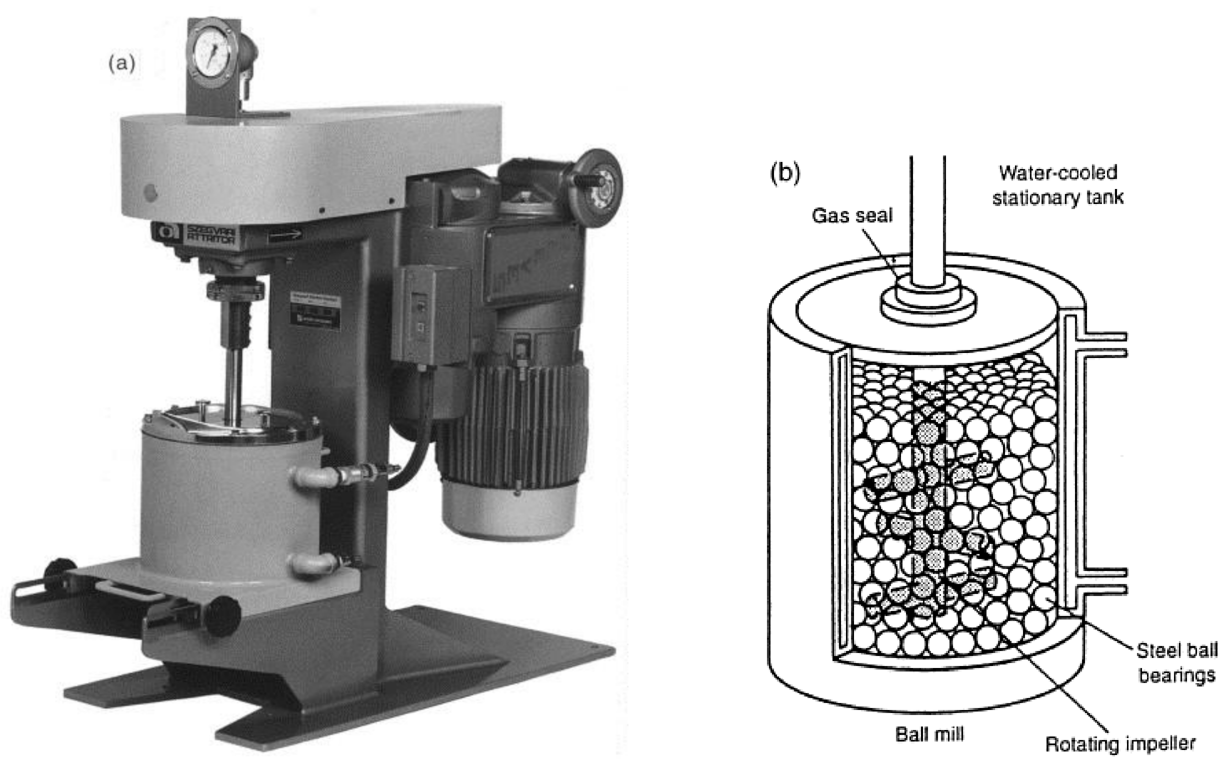


Fig. 4: Attritor Mill [8]

2.3.4 Process Variables:

- Type of mill
- Milling container
- Milling speed
- Milling time
- Type, size, and size distribution of the grinding medium
- Ball-to-powder weight ratio
- Extent of filling the vial
- Milling atmosphere
- Process control agent
- Temperature of milling

Type of mills:

There are a number of different types of mills for conducting MA. These mills differ in their capacity, speed of operation, and their ability to control the operation by varying the temperature of milling and the extent of minimizing the contamination of the powders. Depending on the type of powder, the quantity of the powder, and the final constitution required, a suitable mill can be chosen. Most commonly, however, the SPEX shaker mills are used for alloy screening purposes. Planetary ball mills are used to produce large quantities of the milled powder. Specially designed mills are used for specific applications.

Milling container:

The material used for the milling container (grinding vessel, vial, jar, or bowl are some of the other terms used) is important since due to impact of the grinding medium on the inner walls of the container, some material will be dislodged and get incorporated into the powder. This can contaminate the powder or alter the chemistry of the powder. [4] If the material of the grinding vessel is different from that of the powder, then the powder may be contaminated with the grinding vessel material. On the other hand, if the two materials are the same, then the chemistry may be altered unless proper precautions are taken to compensate for the additional amount of the element incorporated into the powder. Hardened steel, tool steel, hardened chromium steel, tempered steel, stainless steel, WC–Co, WC-lined steel, and bearing steel are the most common types of materials used for the grinding vessels. Some specific materials are used for specialized purposes; these include copper, titanium, sintered corundum, yttria-stabilized zirconia (YSZ), partially stabilized zirconia + yttria and, sapphire and, agate, and, hard porcelain, Si_3N_4, and Cu–Be. The shape of the container also seems to be important, especially the internal design of the container. Both flat-ended and round-ended SPEX mill containers have been used. Alloying was found to occur at significantly higher rates in the flat-ended vial than in the round-ended container.

Milling speed:

It is easy to realize that the faster the mill rotates the higher would be the energy input into the powder. But, depending on the design of the mill there are certain limitations to the maximum speed that could be employed. For example, in a conventional ball mill increasing the speed of rotation will increase the speed with which the balls move. Above a critical speed, the balls will be pinned to the inner walls of the vial and do not fall down to exert any impact force. Therefore, the maximum speed should be just below this critical value so that the balls fall down from the maximum height to produce the maximum collision energy.

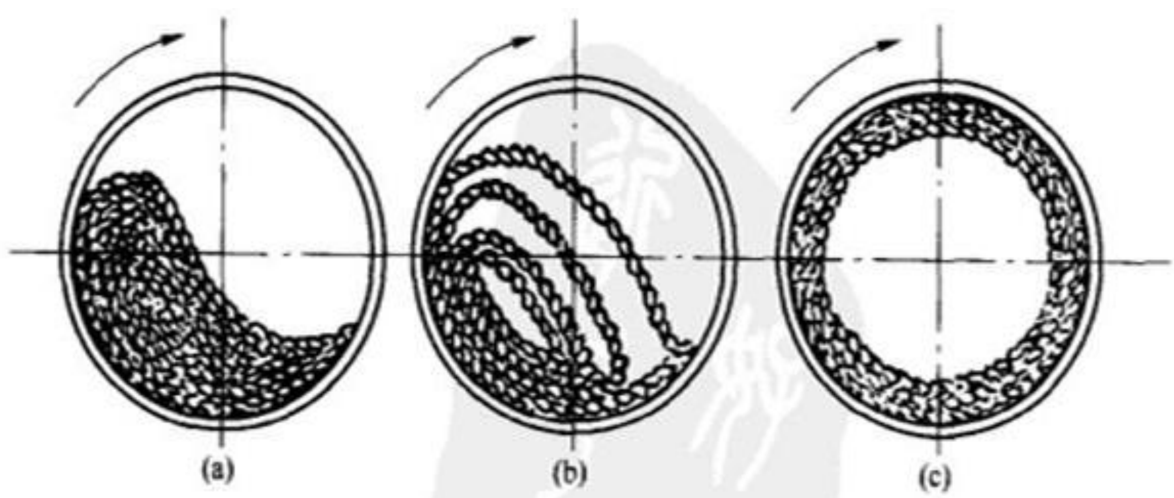


Fig. 5: Ball starts pinning to the inner wall of the vial as speed of rotation increases, and at critical speed of rotation the balls will be completely pinned to the inner walls of the vial and do not fall down to exert any impact force.[6]

Another limitation to the maximum speed is that at high speeds (or intensity of milling), the temperature of the vial may reach a high value. This may be advantageous in some cases where diffusion is required to promote homogenization and/or alloying in the powders. But, in some cases, this increase in temperature may be a disadvantage because the increased temperature accelerates the transformation process and results in the decomposition of supersaturated solid solutions or other metastable phases formed during milling. Additionally, the high temperatures generated may also contaminate the powders.

Milling time:

The time of milling is the most important parameter. Normally the time is so chosen as to achieve a steady state between the fracturing and cold welding of the powder particles. The milling time required varies depending on the type of mill used, the intensity of milling, the ball-to-powder ratio, and the temperature of milling. But, it should be realized that the level of contamination increases and some undesirable phases form if the powder is milled for times longer than required. Therefore, it is desirable that the powder is milled just for the required duration and not any longer [8].

Grinding medium:

Hardened steel, tool steel, hardened chromium steel, tempered steel, stainless steel, WC–Co, and bearing steel are the most common types of materials used for the grinding medium.

The density of the grinding medium should be high enough so that the balls create enough impact force on the powder. The size of the grinding medium also has an influence on the milling efficiency. A large size (and high density) of the grinding medium is useful since the larger weight of the balls will transfer more impact energy to the powder particles.

Ball-to-powder weight ratio:

The ratio of the weight of the balls to the powder (BPR), sometimes referred to as charge ratio (CR), is an important variable in the milling process. This has been varied by different investigators from a value as low as 1:1 to as high as 220:1. A ratio of 10:1 is most commonly used while milling the powder in a small capacity mill such as a SPEX mill. But, when milling is conducted in a large capacity mill, like an attritor, a higher BPR of up to 50:1 or even 100:1 is used [4] and [8].

The BPR has a significant effect on the time required to achieve a particular phase in the powder being milled. The higher the BPR, the shorter is the time required. At a high BPR, because of an increase in the weight proportion of the balls, the number of collisions per unit time increases and consequently more energy is transferred to the powder particles and so alloying takes place faster.

Extent of filling the vial:

Since alloying among the powder particles occurs due to the impact forces exerted on them, it is necessary that there is enough space for the balls and the powder particles to move around freely in the milling container. Therefore, the extent of filling the vial with the powder and the balls is important. If the quantity of the balls and the powder is very small, then the production rate is very small. On the other hand, if the quantity is large, then there is not enough space for the balls to move around and so the energy of the impact is less. Thus, care has to be taken not to overfill the vial; generally about 50% of the vial space is left empty.

Milling atmosphere:

The major effect of the milling atmosphere is on the contamination of the powder. Therefore, the powders are milled in containers that have been either evacuated or filled with an inert gas such as argon or helium. High-purity argon is the most common ambient to prevent oxidation and/or contamination of the powder. [2]

Different atmospheres have been used during milling for specific purposes. Nitrogen or ammonia atmospheres have been used to produce nitrides. Hydrogen atmosphere was used to produce hydrides. The presence of air in the vial has been shown to produce oxides and nitrides in the powder, especially if the powders are reactive in nature. Thus, care has to be taken to use an inert atmosphere during milling.

Process control agents:

The powder particles get cold-welded to each other, especially if they are ductile, due to the heavy plastic deformation experienced by them during milling. But, true alloying among powder particles can occur only when a balance is maintained between cold welding and fracturing of particles. A process control agent (PCA) (also referred to as lubricant or surfactant) is added to the powder mixture during milling to reduce the effect of cold welding. The PCAs can be solids, liquids, or gases. They are mostly, but not necessarily, organic compounds, which act as surface-active agents. The PCA adsorbs on the surface of the powder particles and minimizes cold welding between powder particles and thereby inhibits agglomeration. The surface-active agents adsorbed on particle surfaces interfere with cold welding and lower the surface tension of the solid material. Since the energy required for the physical process of size reduction, *E* is given by

Equation

$$E = \gamma \cdot \Delta S$$

Where γ is the specific surface energy and ΔS is the increase of surface area, a reduction in surface energy results in the use of shorter milling times and/or generation of finer powders. [8]

Temperature of milling:

The temperature of milling is another important parameter in deciding the constitution of the milled powder. Since diffusion processes are involved in the formation of alloy phases irrespective of whether the final product phase is a solid solution, intermetallic, nanostructure, or an amorphous phase, it is expected that the temperature of milling will have a significant effect in any alloy system.

2.4 Mechanism of Mechanical Alloying

During high-energy milling the powder particles are repeatedly flattened, cold welded, fractured and re-welded. Whenever two steel balls collide, some amount of powder is trapped in between them. Typically, around 1000 particles with an aggregate weight of about 0.2 mg are trapped during each collision. The force of the impact plastically deforms the powder particles leading to work hardening and fracture. The new surfaces created to enable the particles to weld together and this leads to an increase in particle size.

Since in the early stages of milling, the particles are soft (if we are using either ductile-ductile or ductile-brittle material combination), their tendency to weld together and form large particles are high. A broad range of particle sizes develops, with some as large as three times bigger than the starting particles. The composite particles at this stage have a characteristic layered structure consisting of various combinations of the starting constituents. With continued deformation, the particles get work hardened and fracture by a fatigue failure mechanism and/or by the fragmentation of fragile flakes. Fragments generated by this mechanism may continue to reduce in size in the absence of strong agglomerating forces. At this stage, the tendency to fracture predominates over cold welding. Due to the continued impact of grinding balls, the structure of the particles is steady, but the particle size continues to be the same. Consequently, the inter-layer spacing decreases and the number of layers in a particle increase [8].

After milling for a certain length of time, steady-state equilibrium is attained when a balance is achieved between the rate of welding, which tends to increase the average particle size, and the rate of fracturing, which tend to decrease the average composite particle size. Smaller particles are able to withstand deformation without fracturing and tend to be welded into larger pieces, with an overall tendency to drive both very ®ne and very large particles towards an intermediate size. At this stage each particle contains substantially all of the starting ingredients, in the proportion they were mixed together and the particles reach saturation hardness due to the accumulation of strain energy. The particle size distribution at this stage is narrow, because particles larger than average are reduced in size at the same rate that fragments smaller than average grow through agglomeration of smaller particles.

3. OBJECTIVES

We deliberate to:

- Dry and wet grinding behavior of iron (Fe) and sand powder by high energy planetary milling at different milling time up to 2 hours.

- Particle size measurement and phase study during milling.

4. EXPERIMENTAL DETAILS

Planetary milling of iron and sand powder were carried out using a planetary ball mill with hardened chrome steel container and balls. The total weight of balls (8 mm diameter) used was 800gm and a ball to powder weight ratio of 4:1 was used. The jar speed and the supporting main shaft were 620 and 275 rpm respectively. The milling was performed and powders were picked up from the mill at the intervals of 0, 0.5, 1, 1.5 and 2 hours. Toluene was used for wet milling.
The as milled powder was characterized by X-ray diffraction (XRD) with Cu Kα radiation to identify the phases present and particles size was measured by Malvern laser particle size analyser.

Table-1 Different milling parameters used:

Parameter	Value
Balls/Powder weight ratio	4:1
Ball diameter	8mm
Atmosphere	Air
Grinding medium	Dry and wet(toluene)
Weight of ball	200gm.
Milling time	2h (pause mode every 30 min.)
Type of mill	Dual Drive Planetary mill

5. RESULTS AND DISCUSSIONS

5.1 X-Ray Diffraction Analysis

For dry milling (when particles are milled in dry conditions)

Fig. 6 and 7 show the XRD spectra of iron and sand powder milled for different time period in dry conditions.

In Fig. 6, we can see the XRD spectra of iron powder milled for different time periods. Here the peak intensity remains same while the peak width is increasing with increase in milling time. Increase in peak width is due to refinement of powder or reduction in particle size and stress developed during milling. The ball-powder-jar surface collision results in high stress into powder particles.

In Fig. 7, we can see the XRD spectra of quartz powder milled for different time periods. The increase in peak width is prominent in silica. Here although particle refinement takes place but due to brittle nature of sand high stress is not developed into sand particles. Only fracturing of particle takes place.

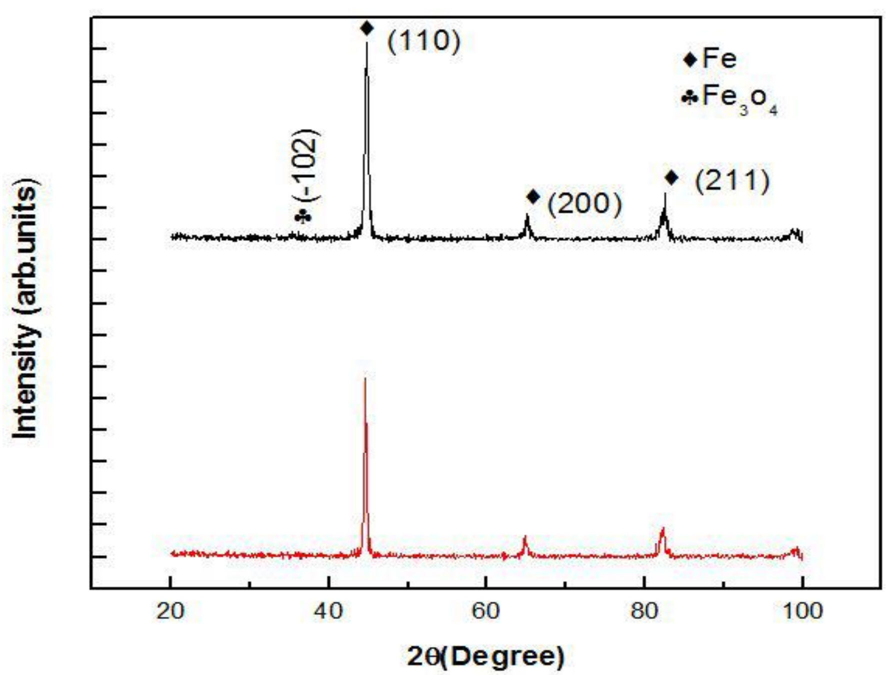


Fig. 6: XRD results of Fe powder at different milling hours

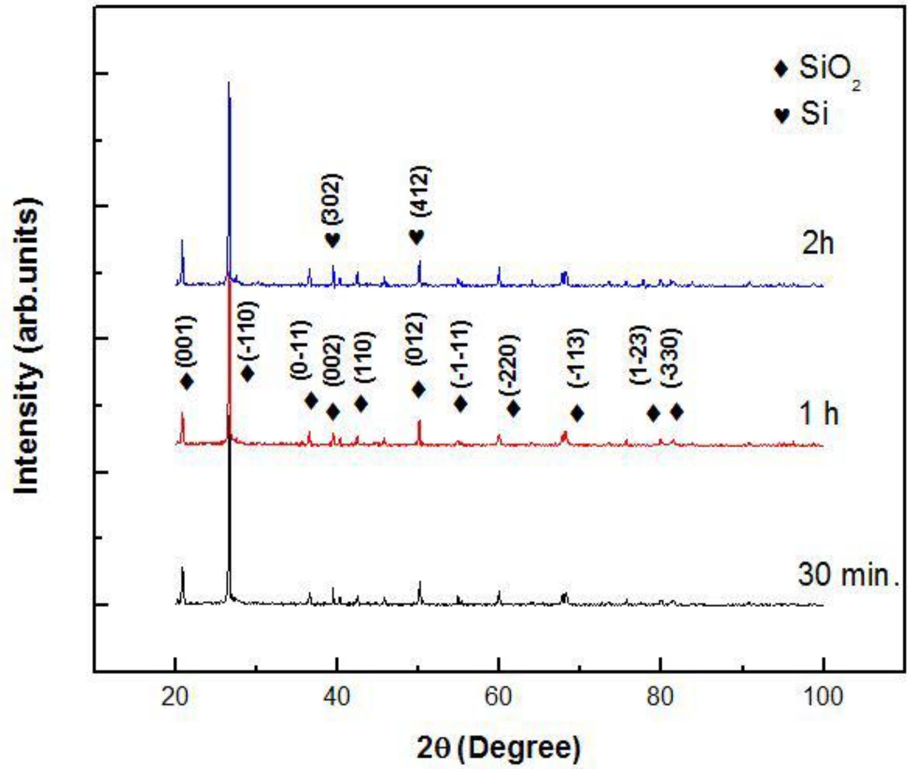


Fig. 7: XRD results of SiO_2 powder at different milling hours

For wet milling (when particles are milled in wet conditions)

Fig. 8 and 9 show the XRD spectra of iron and sand powder milled for different time period in wet conditions.

In Fig. 8, we can see the XRD spectra of iron powder milled for different time periods. **Here the peak intensity is decreasing and also the peak width is increasing unlike in dry milling where the peak intensity remains same while the peak width is increasing with increase in milling time.** Increase in peak width is due to refinement of powder or reduction in particle size and stress developed during milling. The ball-powder-jar surface collision results in high stress into powder particles. **The decrease in peak intensity with increase in milling time is due to more refinement of powder particle (finer powder particle) during wet milling than dry milling.**

In Fig. 9, we can see the XRD spectra of quartz powder milled for different time periods. Here too, the increase in peak width is prominent, but the peak intensity decreases with time. Due to the brittle nature of sand high stress is not developed into sand particles, only fracturing of particle takes place, but as more refinement of particle takes place during wet milling the peak intensity decreases.

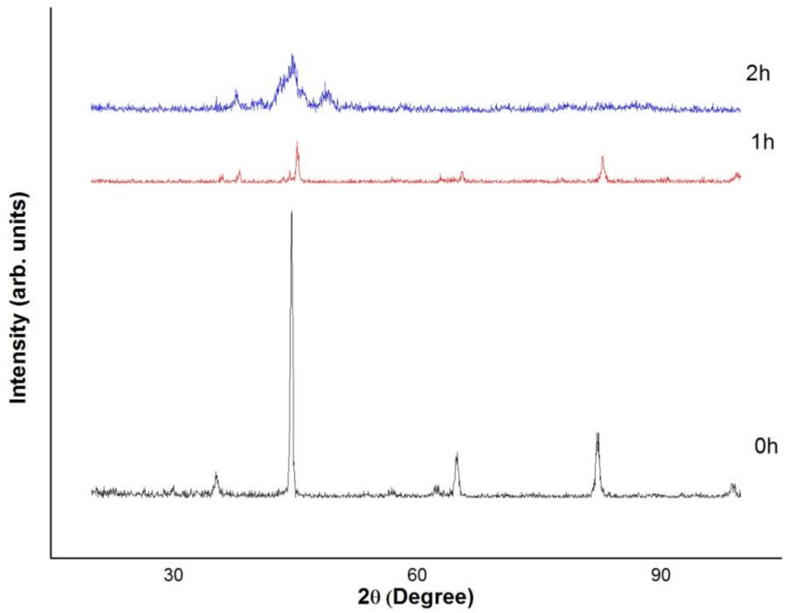


Fig. 8: XRD results of Fe powder at different milling hours

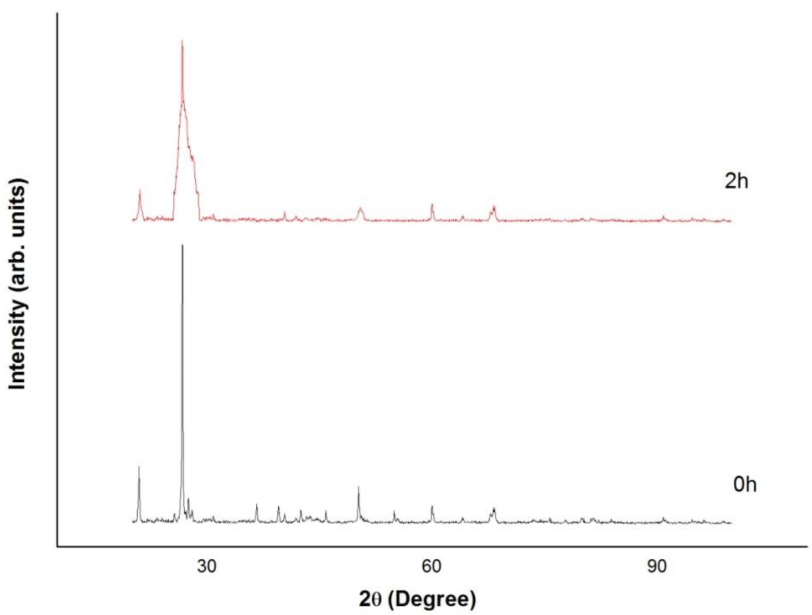


Fig. 9: XRD results of SiO2 powder at different milling hours

5.2 Particle Size Analysis

For dry milling (when particles are milled in dry conditions)

Fig. 10 and 11 shows the particle size distribution of iron powder milled for different time in dry conditions.

It can be seen from graph that particle size distribution is binomial distribution and also size distribution is narrow as compared to sand. Average particle size reduces from 23.90µm to 22.864µm as milling time increases from 0.5 h to 1.5 h.

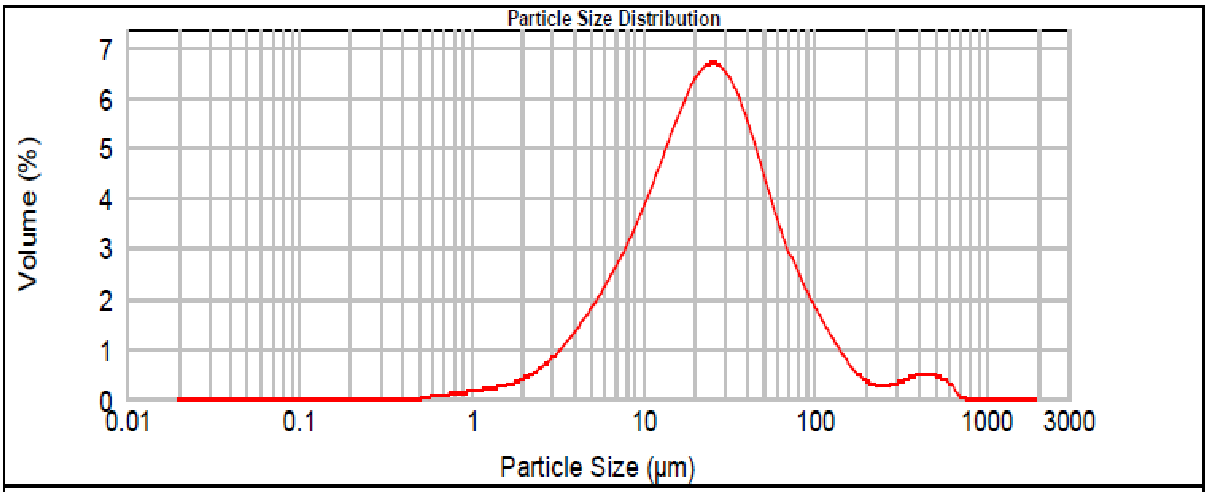


Fig. 10: Particle size distribution of iron at 0.5 h milling time. (avg. 23.90µm)

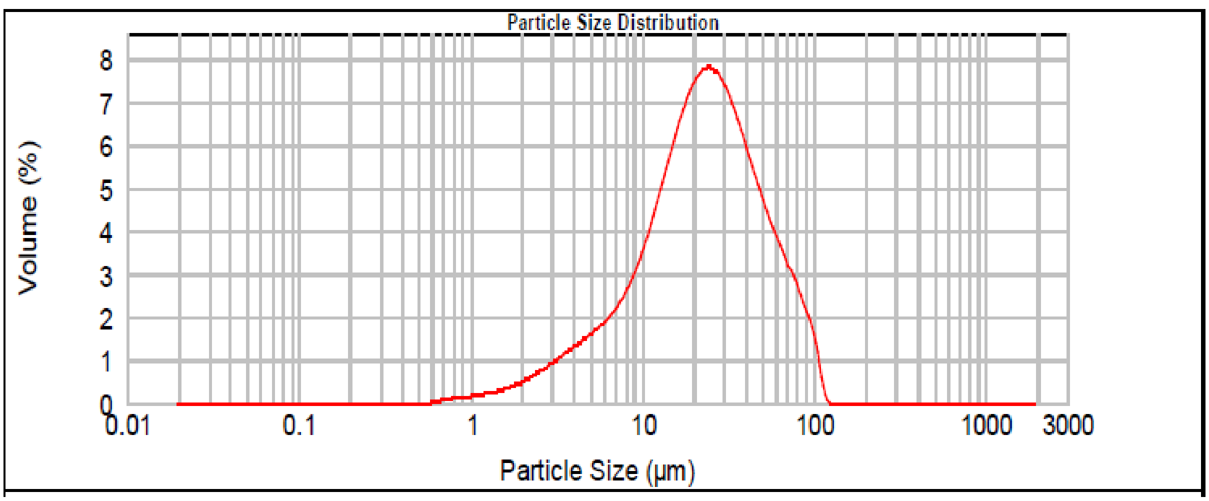


Fig. 11: Particle size distribution of iron at 1.5 h milling time. (avg. 22.864µm)

Fig 12 and 13 shows the particle size distribution of sand powder milled for different time in dry conditions.

The particle size graph shows wide size distribution. It is evident from the figure that the average particle size reduces from 15.097µm to 9.245µm as milling time increases from 0.5 h to 1.5 h.

It is obvious that rate of particle size reduction is very low in case of iron as compared to sand due to ductile nature. During milling ductile iron cold weld together and flattened where as in case of sand, sand particles are fractured or breakages take place due to brittle nature.

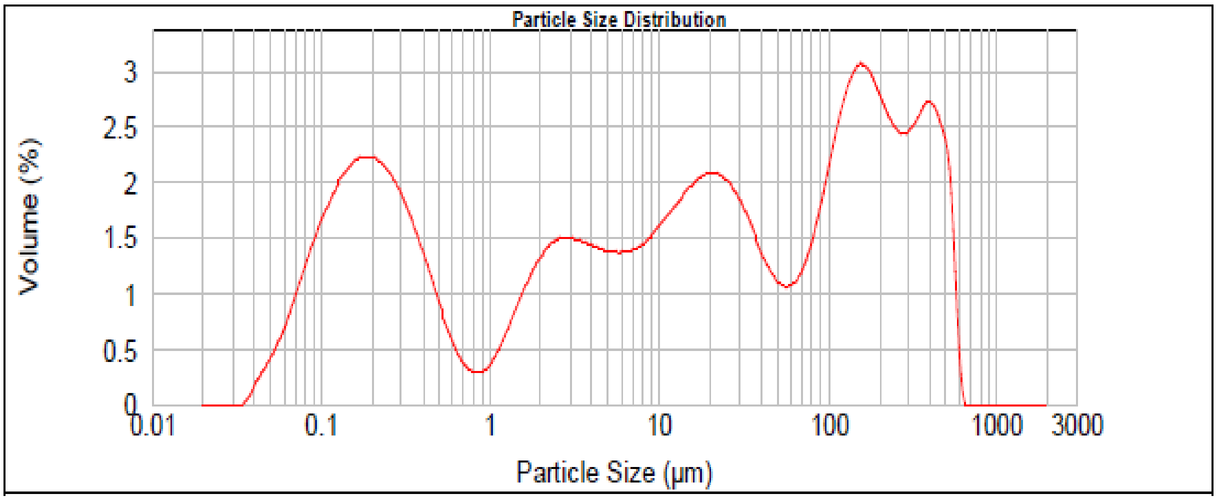


Fig. 12: Particle size distribution of sand at 0.5 h milling time. (avg. 15.097µm)

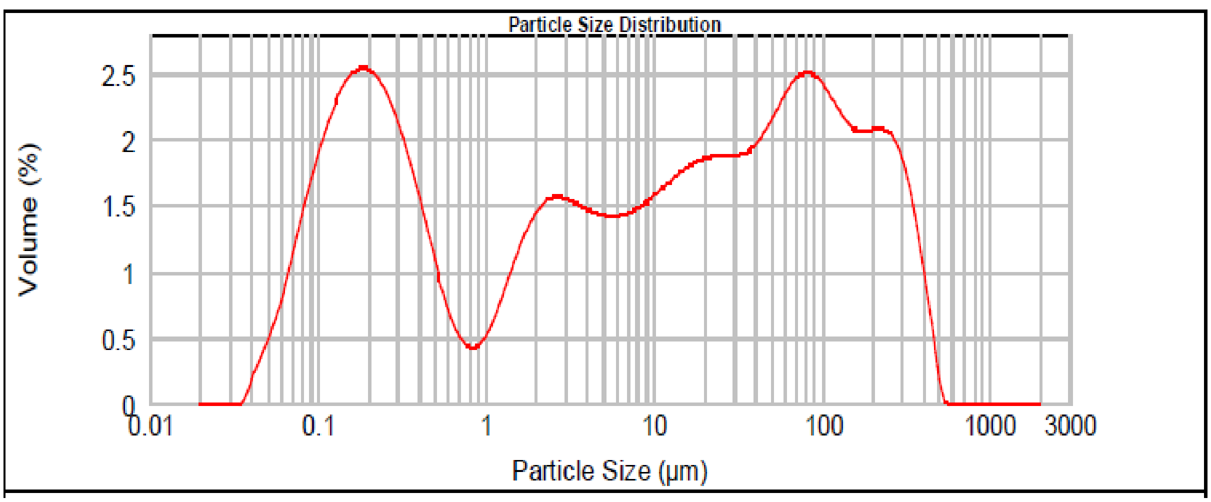


Fig. 13: Particle size distribution of sand at 1.5 h milling time. (avg. 9.245µm)

For wet milling (when particles are milled in wet conditions)

Fig. 14 and 15 shows the particle size distribution of iron powder milled for different time in wet conditions.

It can be seen from graph that particle size distribution is binomial distribution. Average particle size reduces from 1270nm to 1201nm as milling time increases from 0.5 h to 2 h.

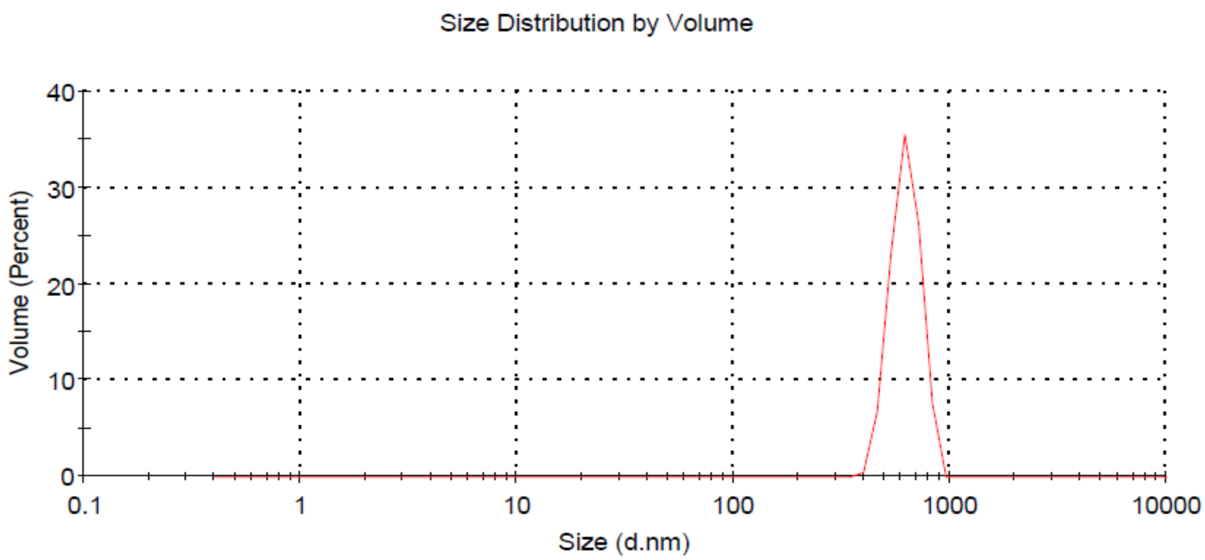


Fig. 14: Particle size distribution of iron at 0.5 h milling time. (avg. 1270nm)

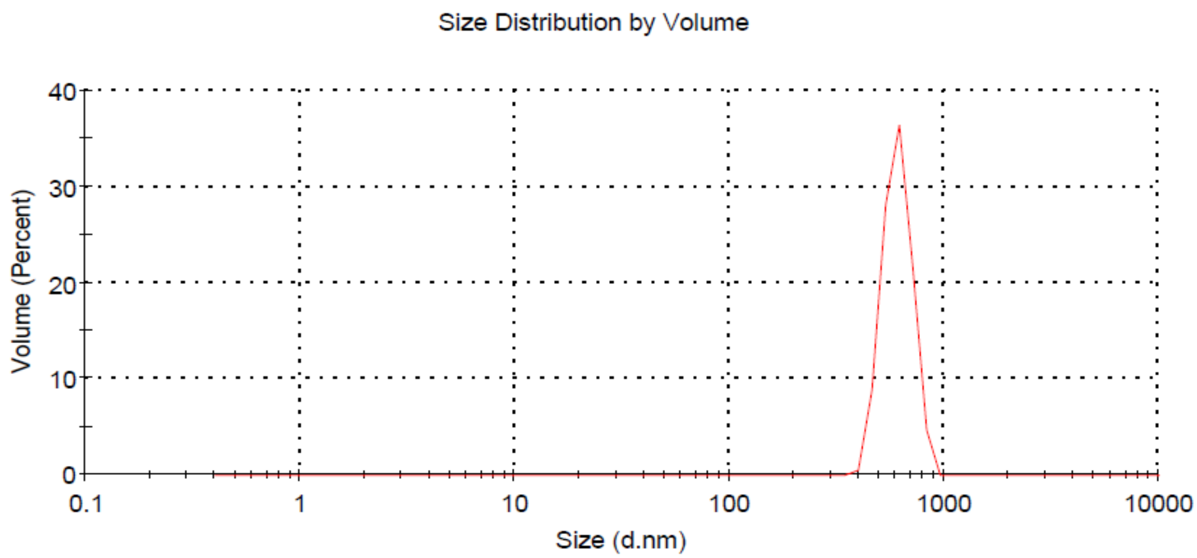


Fig. 15: Particle size distribution of iron at 2 h milling time. (avg. 1201nm)

Fig 16 and 17 shows the particle size distribution of sand powder milled for different time in wet conditions.

The particle size graph shows binomial distribution. It is evident from the figure that the average particle size reduces from 1438nm to 833.2nm as milling time increases from 0.5 h to 2 h.

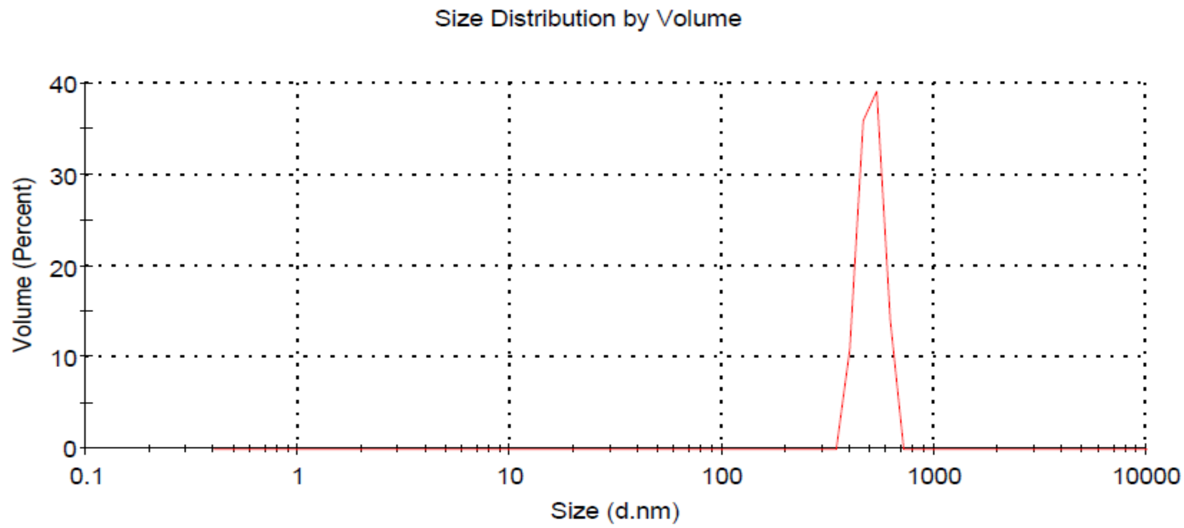


Fig. 16: Particle size distribution of sand at 0.5 h milling time. (avg. 1438nm)

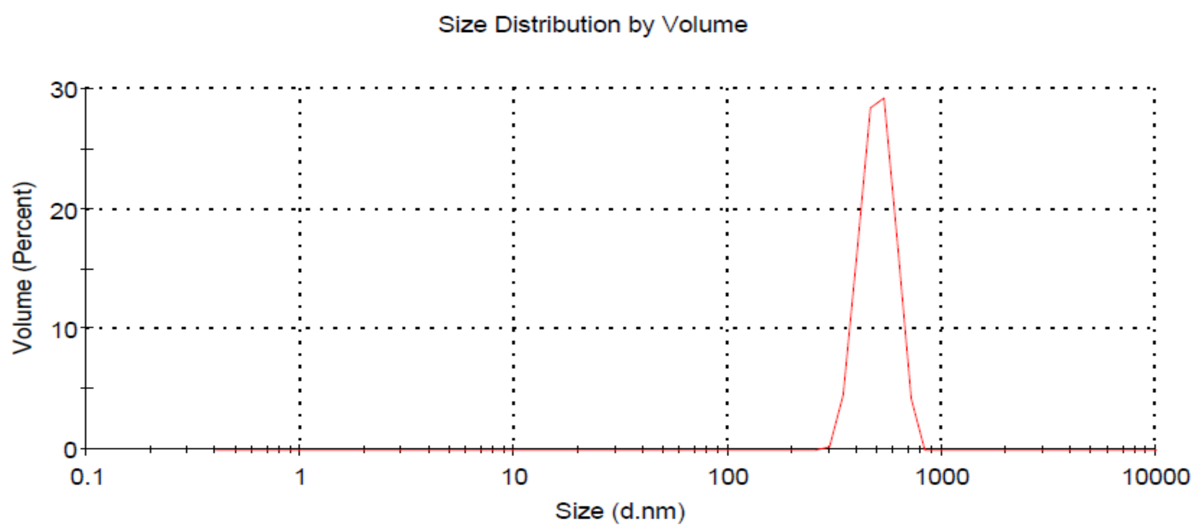


Fig. 17: Particle size distribution of sand at 2 h milling time. (avg. 833.2nm)

Here too, the rate of particle size reduction is very low in case of iron as compared to sand due to ductile nature of iron. **But from fig. 18 and fig. 19, we can see that the particle size of iron and sand are larger during dry milling than wet milling. These is because the powder particle gets agglomerated in dry milling**

while in wet milling the powder particle get dispersed inside the liquid medium(in this case toluene), resulting in finer powder particle.

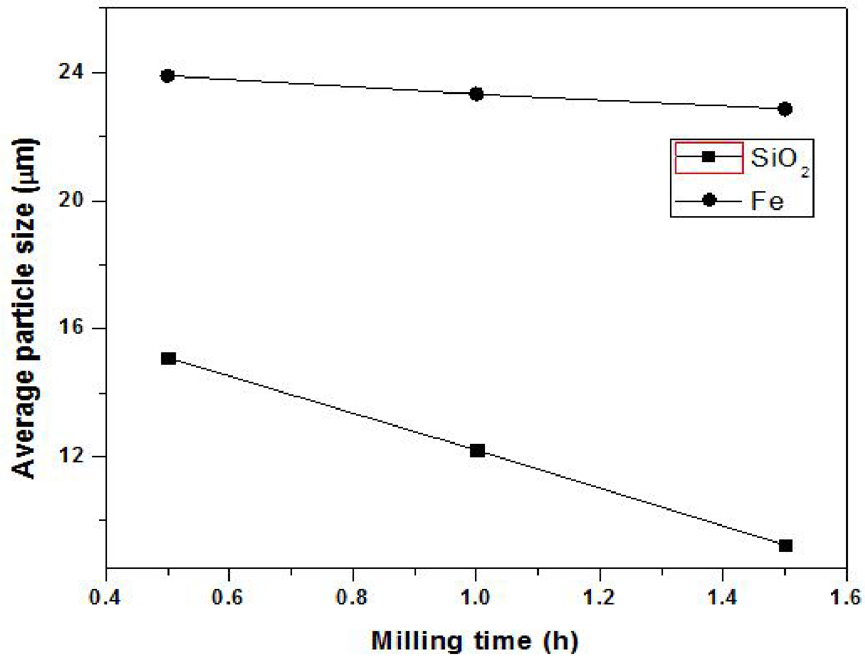


Fig. 18: Graph showing average particle size distribution vs. milling time (when milled in dry conditions)

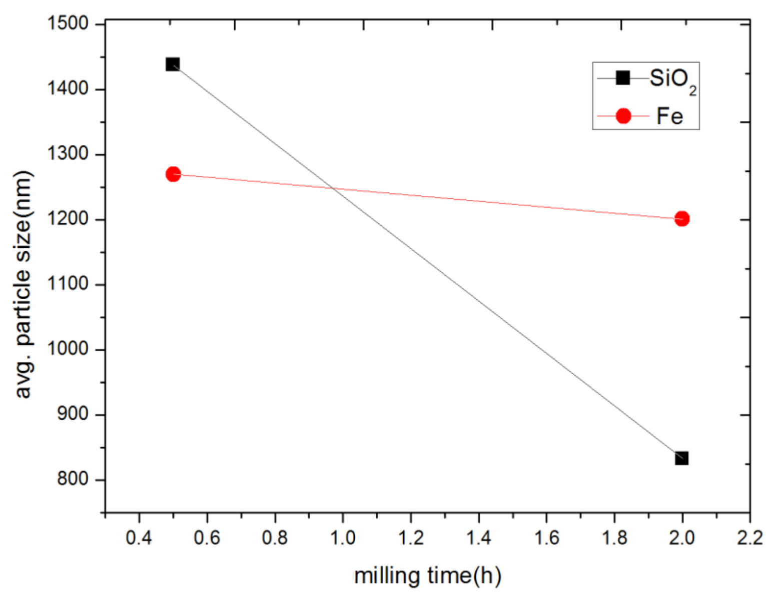


Fig. 19: Graph showing average particle size distribution vs. milling time (when milled in wet conditions)

6. Conclusion

The important conclusions that can be drawn from the results presented here are as follows:

- There is no phase change during dry grinding of iron and sand for 2 h.
- In XRD, the peak intensityof the iron and sand remains same during dry grinding while it decreases during wet grinding, is due to the formation of finer particles in wet milling.
- Average particle size reduces as milling time increases.
- The rate of size reduction is very fast in case of sand as compared to iron due to brittle nature of sand.
- Less fine particles are formed during dry milling than wet milling due to the formation of agglomerates.

7. Future Scope

This project can be further carried out to get more results. Some of the future scope of this project includes:

- Grinding kinetics can be studied for both wet and dry.
- Milling parameters can be varied such as ball-to-weight ratio, milling atmosphere and milling speed, etc. to get further details about the properties of ductile and brittle materials in dry and wet condition.

8. References

[1] C. Suryanarayana (Ed.), Non-equilibrium Processing of Materials, Pergamon Press, Oxford, 1999.

[2] B.S. Murty, S. Ranganathan, Int. Mater. Rev. (1998).

[3] M.O. Lai, L. Lu, Mechanical Alloying, 1998.

[4] C. Suryanarayana, Prog. Mater. Sci. 46 (2000).

[5] Turker M, Karatas C, Saritas S. Computation of flow behaviors of mechanically alloyed and turbola processed powder PIM feedstock by capillar rheometer.

[6] Kilinc Y, Turker M, Saritas S. Investigation of the mechanical properties of iron based super alloys produced by mechanical alloying techniques

[7] H. Arik, Production and characterization of In situ Al4C3 reinforced aluminium-based composite produced by the mechanical alloying technique, Mater Design, 25 (2004).

[8] C. Suryanarayana, Mechanical alloying and milling (2001)