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

A THESIS SUBMITTED IN PARTIAL FULLFILLMENT

OF THE REQUIREMENT FOR THE DEGREE OF

Bachelor of Technology

In

Metallurgical and Materials Engineering

By

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Batch (2010-2014)

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Certificate

This is to certify that the thesis entitled "**Dry Grinding Behavior of Fe and SiO_2 in Specially Designed Dual Drive Planetary Ball Mill**" being submitted by Sunil Kumar (110MM0360) , Arbaz Khan (110MM0573) and Ankit Kumar Pandey (110MM0368) for the partial fulfillment of the requirements of Bachelor of Technology degree in Metallurgical and Materials Engineering is a bonafide thesis work done by them under my supervision during the academic year 2013 - 2014, 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: 8/5/2014

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Abstract

Mechanical alloying (MA) is a solid- state powder processing technique involving repeated welding, fracturing, and rewelding of powder particles in high energy ball mill. In the present investigation we have made an attempt to observe the grinding behavior of ductile iron (Fe) and brittle sand materials in dual drive planetary ball mill for 0h, 15 min, 1/2h, 1h, 1.5h & 2 h. 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 as a function of milling time. It has been found that size reduction rate is very low in case of iron at the initial milling period due to ductile nature. In case of sand, the size reduction rate is very fast due to brittle nature.

Keywords:

Mechanical alloying, Dual drive planetary ball mill, X-Ray Diffraction, Particle size analyzer

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1. Introduction

Mechanical alloying was used first in the early 1970s for the production of oxide-dispersion-strengthened (ODS) superalloys. It is now recognized as a versatile technique for the production of a broad range of powders, from amorphous or nanocrystalline to ODS or intermetallicsamong 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 superalloys 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. Microstructurally, 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 superalloy, 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.

Fig. 1 shows the schematic of ball-powder-ball collision during planetary milling.

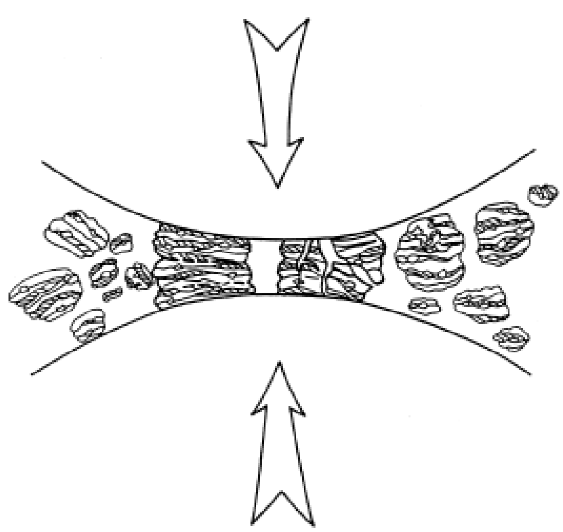


Fig. 1 : Ball-powder-ball collision of powder mixture during reaction milling process

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 intermetallics
- Possibility of alloying of difficult to alloy elements

2.3 Process of Mechanical Alloying

The most important components in MA are as follows:

- Raw Material
- Types of Mills
- 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.

Raw Materials:

Generally raw materials with particle sizes in the range of 1-2000mm 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. Another kind of wet milling is Cryo-milling, 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 dry.

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

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

2.4 Mechanism of Mechnical 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].

3. Objectives

We deliberate to:

- Dry grinding behaviour 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 totall weight of balls(8 mm diameter) was used 800gm and a ball to powder weight ratio of 4:1 were used. Milling was carried out in a specially built dual drive planetary mill. Fig. 2 shows the photograph of the planetary mill. 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.

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 analyzer.

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

Fig. 2 : Photograph of dual drive planetary mill

5. Results and Discussions

5.1 X-Ray Diffraction Analysis

Fig 5.1 and 5.2 shows the XRD spectra of iron and sand powder milled for different time period.

From Fig 5.1,we can see that peak width is increasing with increase in milling time. Increase in peak due to refinement of powder or reduction in particle size and stress developed during milling. The ball powder hyper ball collision results in high stress into powder particles.

In Fig 5.2, shows the XRD spectra of quartz powder milled for different time periods. The increase in peak width is so prominent in silica. Here although particle refinement takes place due to brittle nature of sand but high stress is not developed into sand particles. Only fracturing of particle takes place.

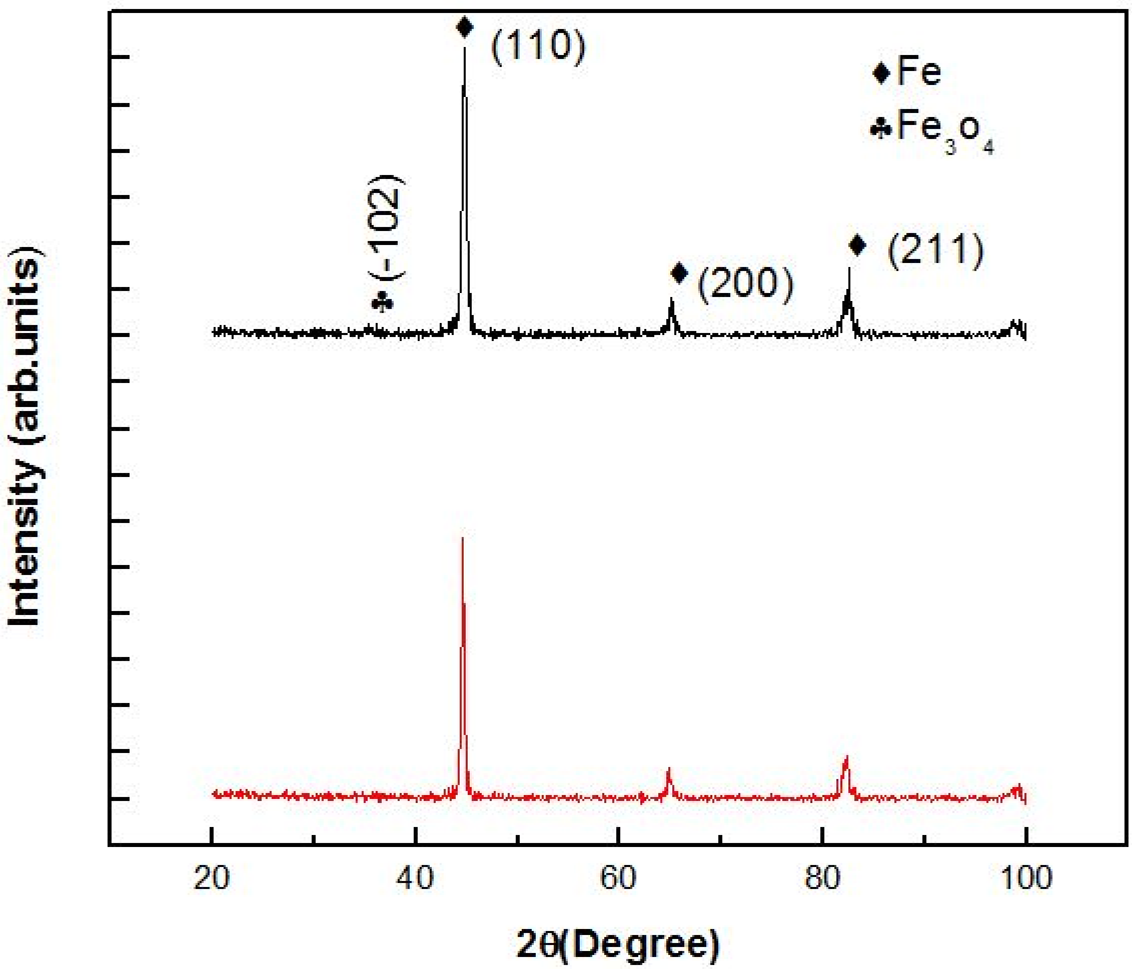


Fig.5.1 : XRD results of Fe powder at different milling hours

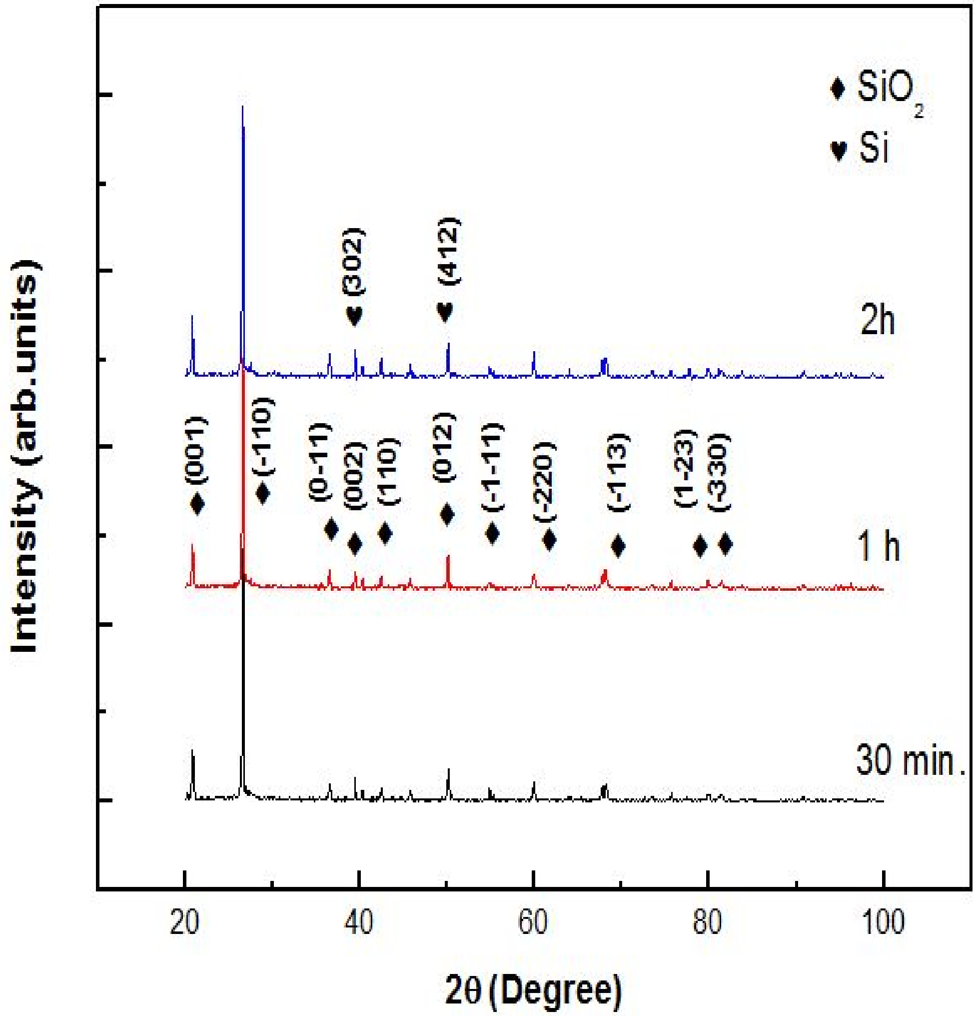


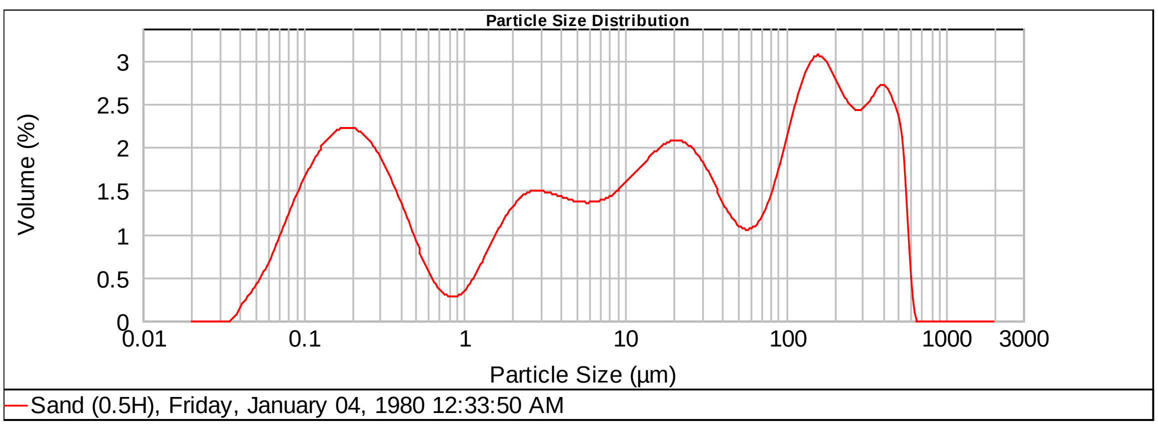
Fig.5.2 : XRD results of SiO_2 powder at different milling hours

5.2 Partical Size Analysis

Fig 5.3 and 5.4 shows the particle size distribution of sand powder milled for 0.5 h and 1.5 h.

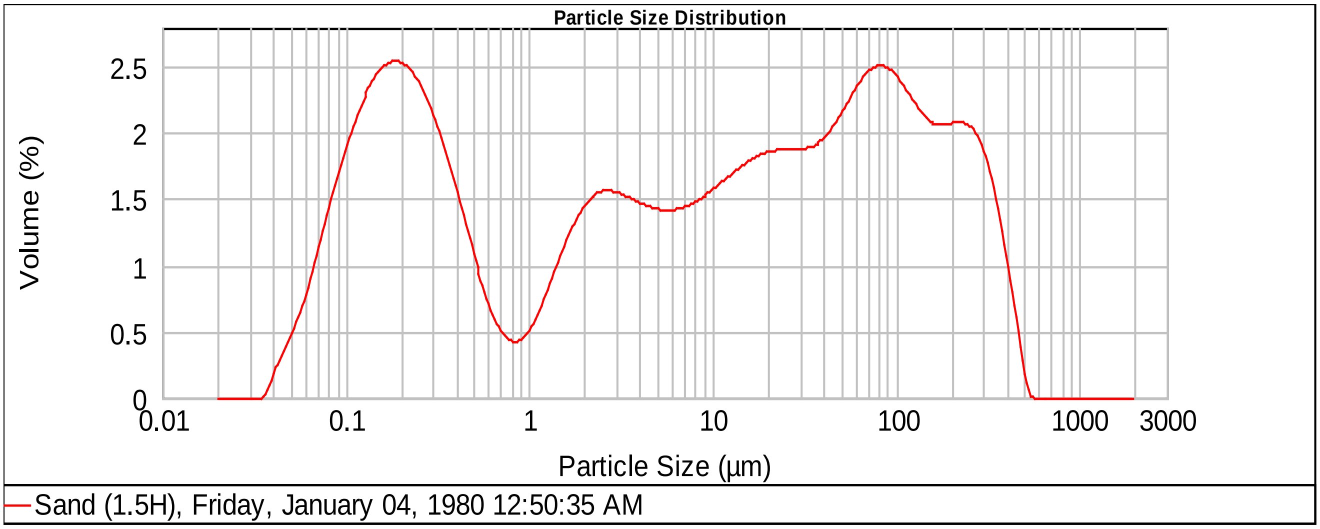
The particle size graph shows wide size distribution. It is evident from the figure the average particle size reduces from 5 to 9 micrometer as milling time increases from 0.5 h to 1.5 h.

Fig 5.5 and 5.6 shows the particle size distribution for iron powder for different time. 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 23.9 micrometer to 22.86 micrometer 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 in 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.



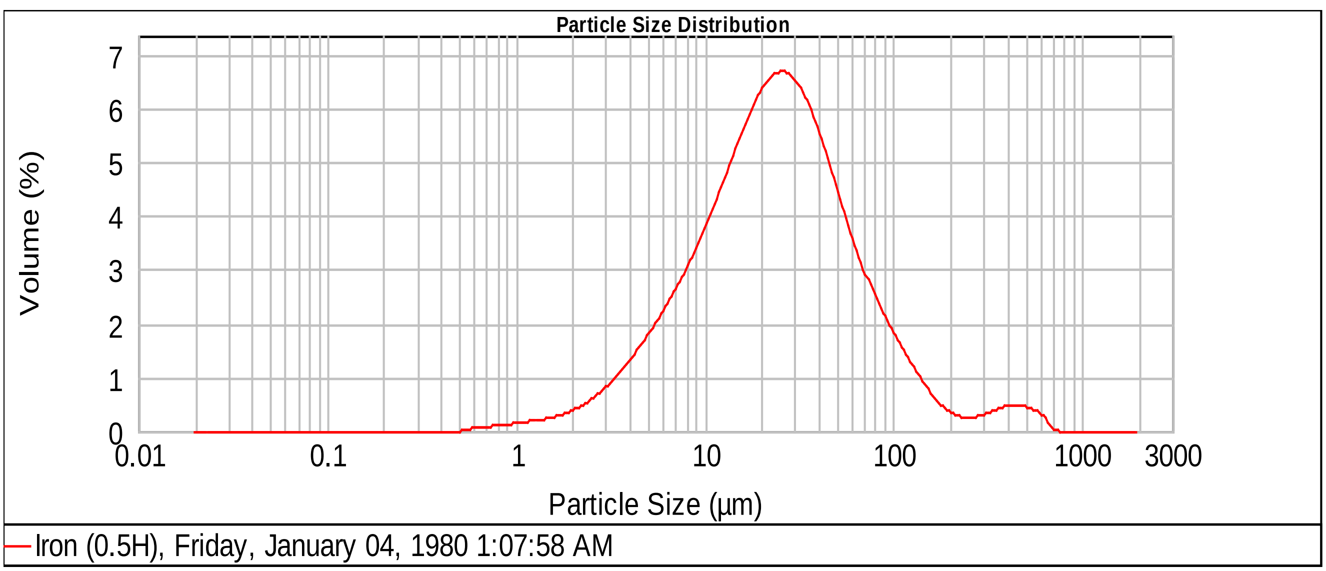
Min 0.151 , Avg 15.097 , Max 311.088 (microns)

Fig.5.3 : Graph showing particle size distribution for sand at 0.5 h milling time.



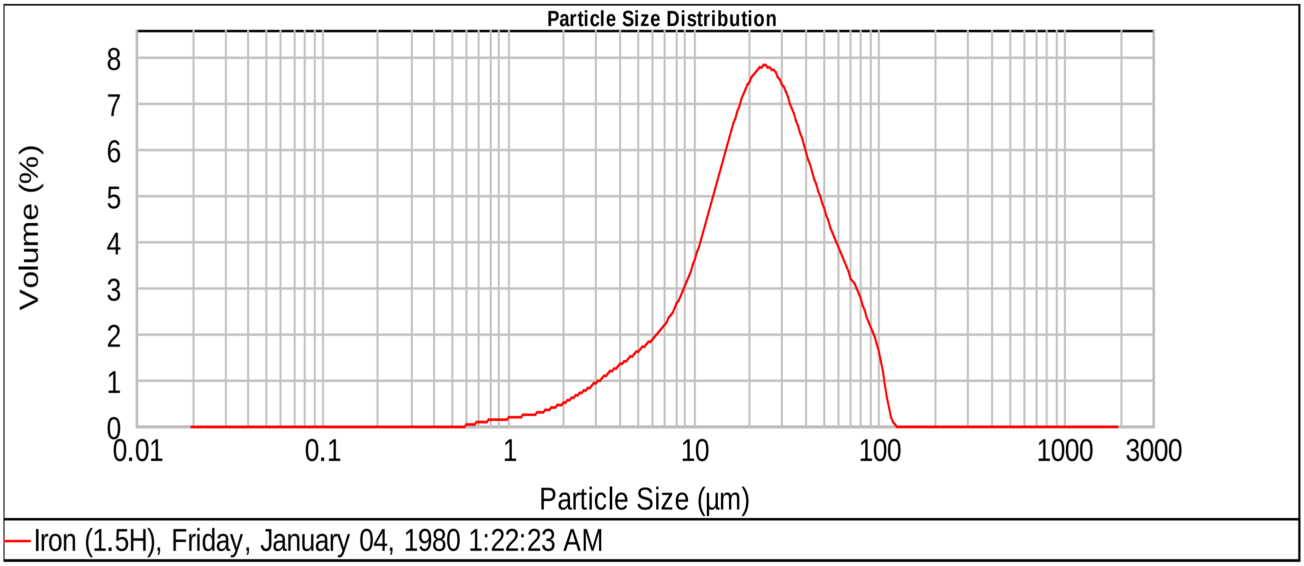
Min 0.137 , Avg 9.245 , Max 188.344 (microns)

Fig.5.4 : Graph showing particle size distribution for sand at 1.5 h milling time.



Min 6.159 , Avg 23.901 , Max 82.023 (microns)

Fig.5.5 : Graph showing particle size distribution for Fe at 0.5 h milling time.



Min 6.122 , Avg 22.864 , Max 60.511 (microns)

Fig.5.6 : Graph showing particle size distribution for Fe at 1.5 h milling time.

Average Particle Size Vs Milling Time:

As we can see from the graph that as time increases particle size decreases for both iron and sand. But size reduction rate is more in sand, due to brittle nature, than iron.

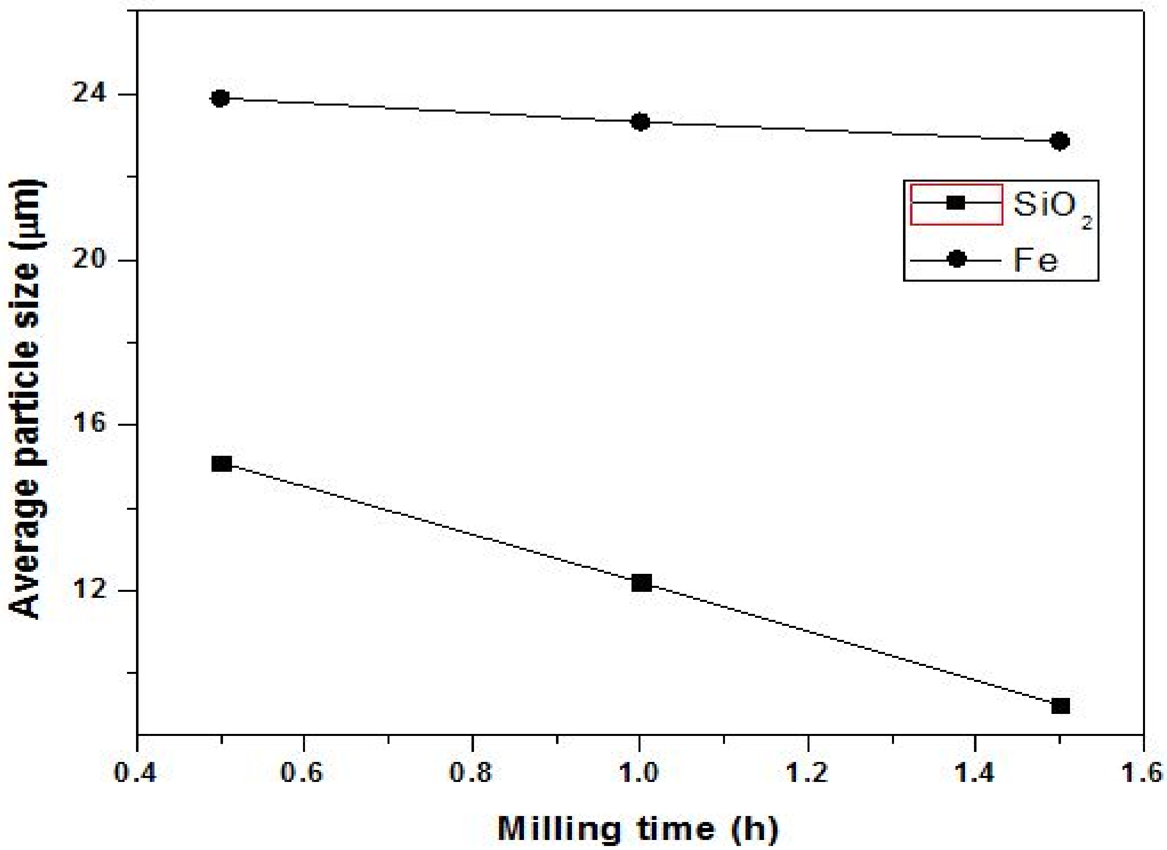


Fig.5.7 : Graph showing average particle size distribution vs milling time

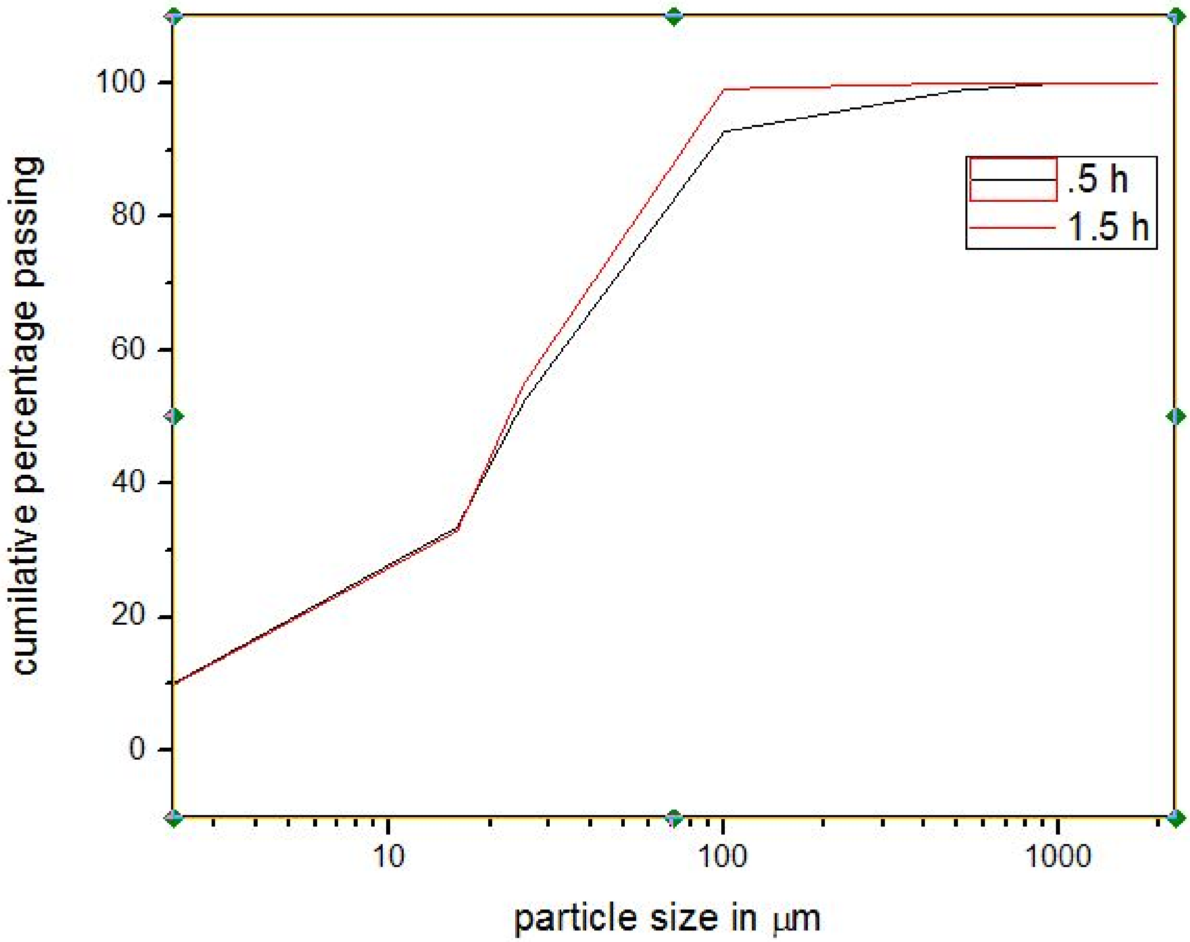


Fig.5.8 : Graph showing particle size distribution for Fe

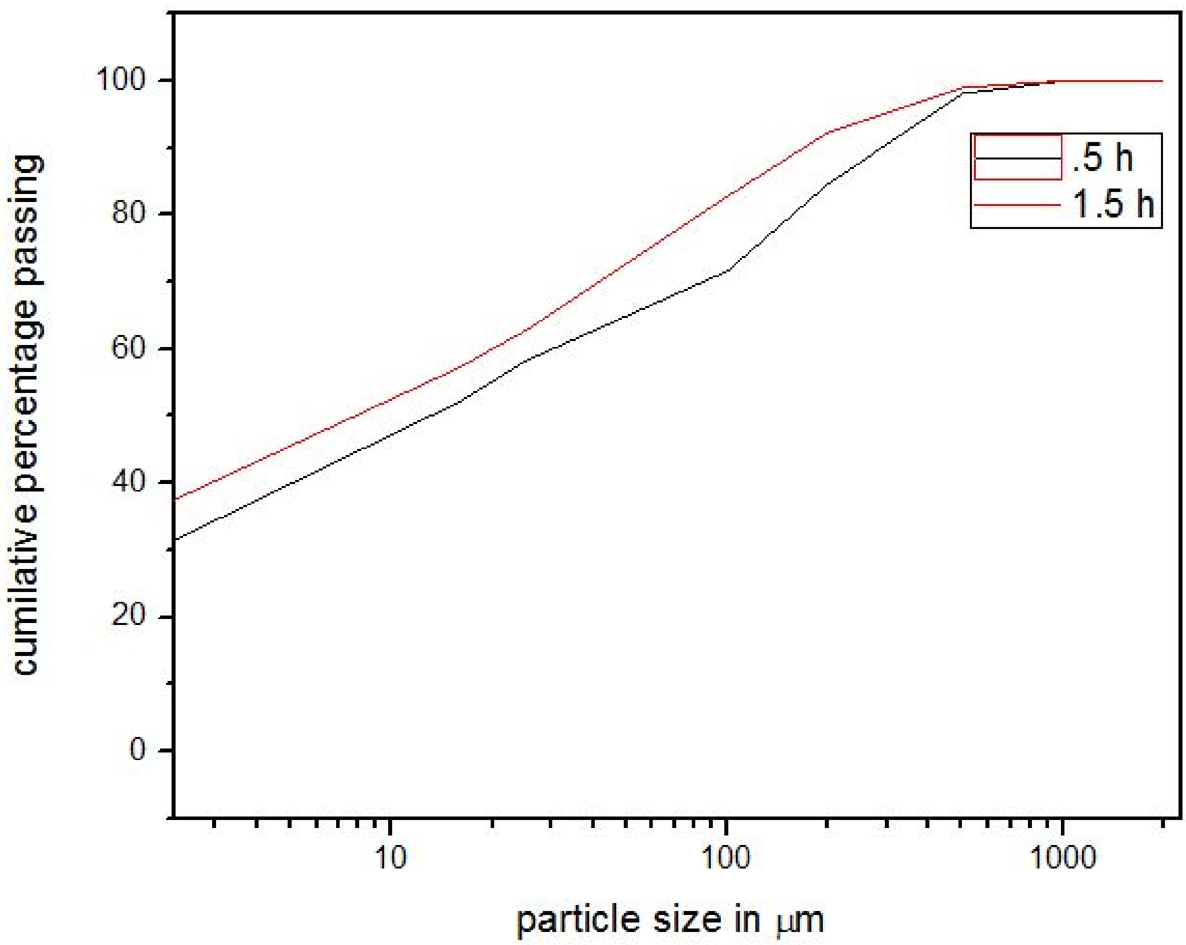


Fig.5.9 : Graph showing particle size distribution for Sand

6. Conclusion

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

- There is no phase change during dry grinding of iron and sand for 2 h.
- The rate of size reduction is very fast in case of sand as compared to iron due to brittle nature of sand.
- The rate of size reduction is very slow in case of iron as compared to sand due to ductile nature of iron.
- Average particle size reduces as milling time increases.

7. Future Scope

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

- Wet milling can be studied.
- 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.

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