

CHARACTERISTICS OF COLD WORKED AL-SI SPRAY FORMED PARTS

A DISSERTATION

*Submitted in partial fulfillment of the
requirements for the award of the degree*

of

MASTER OF TECHNOLOGY

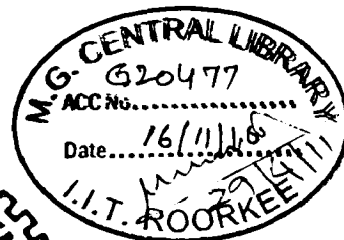
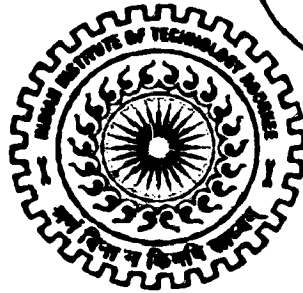
in

METALLURGICAL & MATERIALS ENGINEERING

(With Specialization in Industrial Metallurgy)

By

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JUNE, 2010

CANDIDATE'S DECLARATION

I hereby declare that the work which is being presented in the project entitled "CHARACTERISTICS OF COLD WORKED Al-Si SPRAY FORMED PARTS" in partial fulfillment of the requirement for the award of the degree of Master of Technology in Industrial Metallurgy, submitted in Department of Metallurgical and Materials Engineering, Indian Institute of Technology, Roorkee is an authentic record of my own work carried out for the period from July 2009 to May 2010 under the supervision of **Dr. Devendra Singh**, Assistant Professor at the Department of Metallurgical and Materials Engineering, Indian Institute of Technology Roorkee. The matter embodied in this report has not been submitted by me for the award of any other degree or diploma.

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CERTIFICATE

This is to certify that the above statement made by the candidate is correct to the best of my knowledge.


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ACKNOWLEDGEMENT

I would like to express my deep sense of gratitude to my guide Dr. Devendra Singh, Assistant Professor, Department of Metallurgical and Material Engineering (MMED), I.I.T. Roorkee, for this invaluable guidance, unfailing inspiration and generous help in carrying of this work. This work is simply the reflection of his thoughts, ideas, concepts and about their efforts. Working under their guidance was a privilege and an excellent learning experience that I will cherish for life time.

Author is highly obliged and wishes to owe his sincere thanks to the technical and administrative staff of MMED, especially to Mr. Rajinder S Sharma and Mr. Naresh Kumar. Who helped in all possible ways during the experimental work.

My friends have always been there with their helping hands. I want to thank them for their interest, important hints and suggestions, especially to Mr Kamlesh joshi for providing assistance during experimental work

I can not forget to recall my respected parents and family members.it was the power of their blessing, which gave me courage and confidance to materialize my dreams.

Thanks to all those who have directly and indirectly helped me at any stage of my work



RAJESH RAJPOOT.

ABSTRACT

Al alloys has high strength, low density, good wear resistance, low coefficient of thermal expansion. Its low density, which can contribute significantly to the aspect of weight savings in the design and construction of automotive and aerospace components, materials handling equipment, portable tools and even sporting goods. These properties can be improved by the mechanical processing. By these processes mechanical properties such as ultimate strength, yield strength and percentage elongation and microstructure of the Al alloy may be improved. A rolling thickness reduction of 5% produces Al-Si strip having optimum properties. The mechanism of densification in the deposit layer during rolling has been discussed on the basis of the examination of the microstructure of the deposit at different stages of rolling.

It is observed that the wear behaviour is affected by various mechanical properties. The wear behaviour is also dependent on the load and hardness.

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CHAPTER-1

INTRODUCTION

Aluminum alloys are used in a wide variety of applications because of their attractive properties, such as a high strength to weight ratio, good corrosion resistance, high thermal and electrical conductivity, a low wear rate and good response to various finishing processes. Therefore, these alloys are widely used in automotive and aerospace applications.

Spray forming is a relatively new metallurgical process for the manufacture of near net shaped metallic products with enhanced material properties and performance. The spray formed products are characterized by fine, uniform and macro-segregation free microstructures due to rapid solidification of gas atomized molten droplets during flight and on deposition. Uniform distribution of refined primary silicon particles and modified eutectic phase can be obtained in spray formed hypereutectic aluminum silicon alloys. Oxidation of the alloys is significantly reduced since spray forming is usually conducted in a protective atmosphere in a single-step operation of converting molten alloy directly into a bulk material with specific shape. Therefore, spray forming is a very suitable process for the manufacture of hypereutectic aluminum silicon alloys. Despite many investigations in the field of spray forming hypereutectic Al-Si alloys, systematic study of the influence of processing conditions on the spray formed hypereutectic Al-Si alloys is rarely seen. Some uncertainties remain on how the cooling and solidification conditions in the spray and in the deposit interact to give rise to the various spray formed microstructures.

Silicon is a hard phase and gets embedded in the matrix of Aluminum alloy. It also provides greater hardness and improves wear and seizure resistance. The excellent mechanical and tribological properties of aluminium-silicon alloys have led to extensive use of these alloys in engineering applications, particularly in plain bearings, internal combustion engine pistons and cylinder liners. Although aluminium-silicon alloys meet many of the service requirements, such as high strength-to-weight ratio, excellent corrosion resistance, good bearing qualities and lower expansion characteristics, their poor resistance to seizure makes them vulnerable under poor lubricating conditions, especially during starting or warming-up of engines. In recent years, to overcome this problem, there has been an increasing trend to use solid lubricants over a broad range of

applications . Suitable techniques have been also developed to disperse such solid lubricants in aluminium matrix. However, there is no single material in use which can be regarded as a universal solid lubricant that can perform the desired functions of separating the two moving surfaces under boundary conditions of lubrication and decrease the wear and friction under all operating conditions. Moreover, mere improvements in the tribological properties of Al-Si alloys do not necessarily make them ideal choice for such applications as bearings, pistons or cylinder blocks of internal combustion engines. The materials for such applications should also meet certain physical and mechanical specifications including the ductility and thermal conductivity. Most of the ceramics dispersions used, adversely affect these properties. For example, the thermal conductivity of aluminium alloys decreases with addition of graphite and the latter fails to act as lubricant in vacuum . One of the important mechanical properties such as ductility also decreases with increase in dispersed ceramic particles . The addition of lead to aluminium-silicon alloys can meet many of the above requirements and can act as a solid lubricant to minimize the chances of seizure.

Mechanical processing such as forging rolling and extrusion are suitable for manufacturing low-cost and high quality automotive components in high strength aluminium alloys. This method is particularly suitable for parts with narrow geometrical tolerances, good concentricity, smooth surface finish and for near net shape products. However, an increasing request for producing components at a lower cost requires even more economical production processes. Forming in the warm condition is an alternative process that has the advantages of producing rather complicated geometrical shapes in less operation steps compared to cold forming. In addition, warm forming at moderate temperatures has all the benefits of cold forming including good control of the microstructure and thereby improved strength and ductility.

CHAPTER-2

LITERATURE REVIEW

2.1 SPRAY FORMING

Spray forming, also called spray casting or spray deposition, is the gas atomization of a liquid metal stream into variously sized droplets that are then propelled away from the region of atomization by the fast flowing atomizing gas. The droplet trajectories are interrupted by a substrate which collects and solidifies the droplet into a coherent, near fully dense form. By continuous movement of the substrate relative to the atomizer as deposition proceeds, large forms can be produced in a variety of geometries including billets, tubes and strip.

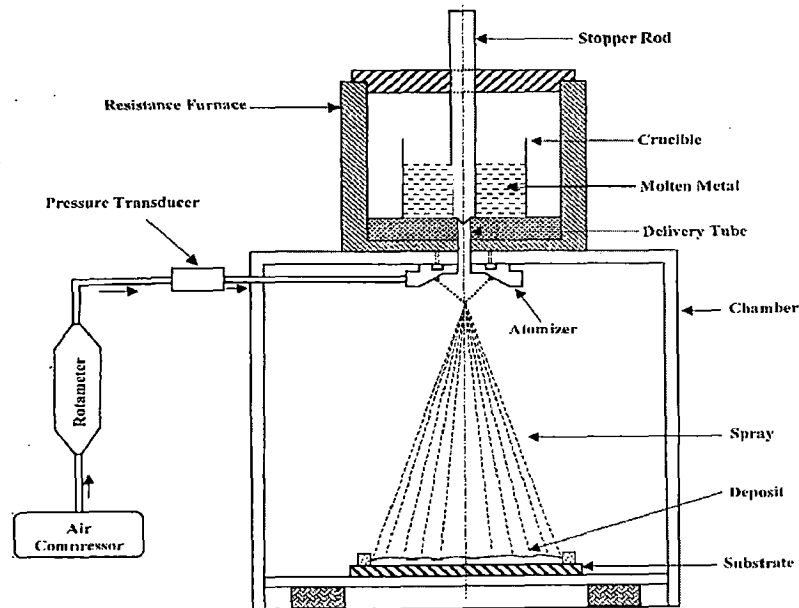


Fig 2.1. Schematic diagram of the spray forming process

The importance of the spray forming technique has been emphasized due to several beneficial effects on processing of composite material by this method. High energy gas jets interact with the stream of molten metal and alloy to generate a spray of micron sized droplets which are directed towards a substrate for deposition.

2.1.1 BASIC PHYSICS INVOLVED IN SPRAY FORMING

The basic physics involved are:

- (1) The fragmentation of a continuous liquid stream into discrete droplets;
- (2) Multi-phase flow of the gas-droplet spray cone and non-linear heat transfer;
- (3) Droplet deposition, splashing and re-deposition; and
- (4) Perform solidification and microstructure evolution.

The multiscale, dynamic and non-linear nature of the inter-related physics of the spray forming process present a significant challenge for the development of meaningful numerical models of the process.

Spray forming or spray deposition is the inert gas atomization of the liquid metal stream which contains variously sized droplets, which are propelled away from the region of atomization by the fast flowing atomization gas. The droplet motion is interrupted by a substrate which collects and solidifies the droplet into a coherent nearly fully dense preform. The schematic of the process is shown FIG 2.1 The droplet formed during atomization are cooled both by convection as well as radiation mode of heat transfer during the flight. Thus these droplets experience high cooling rates. High cooling rate of droplets are also due to increased surface to volume ratio. Since different cooling rates are encountered for various size droplets, they possess unlike thermal states away from the atomization zone. The larger droplets experience slower cooling rate than that of the smaller one which completely solidify due to high cooling rates. Intermediate size droplets remain in a mushy state. These droplets when impinge on to the substrate, get fragmented and this leads to grain multiplication. Entire spray forms a semi-solid/liquid pool on the top surface of the preform. The larger mass fraction is confined to the central region of the preform. Thus the shape of preform depends upon the flat mass flux distribution of droplets in the spray.

2.1.2 HISTORY OF THE SPRAY FORMING

The concept of spray forming has come into existence in early 1970s. Prof. Singer pioneered the spray forming process at Swansea University, Wales, in the early 1970s as the spray rolling process. His work was confined to Al alloys. Later on spray forming was further developed and subsequently licensed by Osprey metals, Neath, Wales, in early 1980s and consequently spray forming is often referred to as the "Osprey process. Further in late 1980s liquid dynamic compaction (LDC) process which is similar to spray forming was developed by Lavernia and Grant. It is worthwhile to point out here that

LDC, Osprey process, and spray forming are the generic names of similar or related processes. The melt in LDC was atomized at a high-gas pressure to generate the maximum yield of small size droplets in the spray. The cooling rate of a large fraction of droplets was well within the rapid solidification regime. Basics of spray forming technology Spray forming being an emerging technology consists Of a very few processing steps than the other emerging technologies like powder metallurgy. This process can be applied to a variety of ferrous and non-ferrous alloys and composites. diameter, some of which, depeSpray forming offers certain advantages over both conventional ingot metallurgy and more specialist techniques such as powder metallurgy. Firstly, it is a flexible process and can be used to manufacture a wide range of materials, some of which are difficult to produce by other methods, e.g. Al-5wt%Li alloys or Al-SiC, Al-Al₂O₃ metal matrix composites (MMCs). The atomization of the melt stream into droplets of 10-500mnding on diameter, cool quickly to the solid and semi-solid state provide a large number of nucleants for the residual liquid fraction of the spray formed material on the billet top surface. The combination of rapid cooling in the spray and the generation of a large population of solid nucleants in the impacting spray leads to a fine equiaxed microstructure, typically in the range 10 - 100 mm, with low levels and short length scales of internal solute partitioning. These microstructural aspects offer advantages in material strength because of fine grain size, refined distribution of dispersoid and / or secondary precipitate phases, as well as tolerance to impurity 'tramp' elements. This fine structure in the 'as sprayed' condition means homogenizing heat treatments can often be avoided. Because of the complex solidification path (i.e. the rapid transition from superheated melt to solid, liquid or semi-solid droplet to temperature equilibration at semi-solid billet top and final slow cooling to fully solid) of the spray formed material, extended solubility of alloying elements and the formation of metastable and quasi- crystalline phases has also been reported.

One of the major attractions of spray forming is the potential economic benefit to be gained from reducing the number of process steps between melt and finished product. Spray forming can be used to produce strip, tube, ring, clad bar / roll and cylindrical extrusion feed stock products, in each case with a relatively fine-scale microstructure even in large cross-sections. The benefits of GASF over powder metallurgy accrue from the reduced number of process steps where powder sieving, pressing, de-gassing and handling steps and their attendant safety and contamination issues may be removed.

2.1.3 BASIC STEPS INVOLVED IN SPRAY FORMING

Spray forming consists of the sequential steps of:

1. Atomization of liquid materials to create a droplet spray;
2. Control of the resulting droplet spray in the gas flow field to achieve a desired droplet mass and enthalpy distribution prior to deposition.
3. The deposition of droplets at a surface to build up a pre-defined preform fig 2.1 shows the spray forming of a preform, indicating the approximate physical regions for the melt atomization, droplet flight and droplet deposition steps. Spray forming is an inherent multiphysics process consisting of multi-length (micrometers to meters) and multi-time scale (microseconds to minutes) transport phenomena.

2.1.4 PROCESS PARAMETERS IN SPRAY FORMING PROCESS

The various process parameters in a spray forming process are as follows:

1. Melt to gas ratio,
2. Flow rate of molten metal,
3. Atomizing gas pressure,
4. Distance between the nozzle and the substrate and the orientation of the substrate.

These parameters play a vital role in refining the microstructure of the preform and thereby enhance the mechanical and wear properties. These independent process parameters can be directly controlled during the process and affect a number of dependent process parameters like the state of spray at deposition and the state of the deposition surface. Therefore, these process parameters should be controlled to produce the optimum mixture of liquid and solid droplets that are essential for producing a quality preform

Therefore, it can be concluded that optimum control of the process parameters is necessary to obtain a required microstructure. There are several process parameters which affects preform integrity which have been identified in the spray atomization and depositions processing as enumerated below.

1. Melt superheat (50-200 °C)
2. Melt flow rate
3. Gas pressure
4. Nozzle to substrate distance

5. Substrate motion, which includes substrate rotation speed, withdrawal rate, tilt angle.

In addition to these there are several pre-set (or design) parameters which can be selected prior to, but not during, the operation of process. These are given below:

1. Melt flow tube diameter
2. The type of atomizing gas
3. Substrate material
4. The gas atomizer design

2.1.5 MERITS, DEMERITS, AND APPLICATIONS OF SPRAY FORMING

Due to cut throat competition in the global market, rapid changes are taking place in the manufacturing sector. High tech industries are coming up with brand new and efficient manufacturing processes. The aim of any such process is to produce a component economically to a near net shape preform in less time. Although the metal casting process produces a component with a reasonably good quality, it lacks in providing a fine grain structure in the cast product severely limiting its application in critical service conditions.

The various benefits that result from spray forming are:

- A near net shape preform,
- Few processing steps,
- Shorter manufacturing time,
- Fine microstructure (uniform distribution of fine grains, no macroscopic segregation of
- Alloying elements, and a low oxide content),
- Better mechanical properties, better wear properties,
- Composites can be produced, different shapes like billets, tubes, strips, and discs can be produced ,
- A wide variety of materials can be applied ranging from non-ferrous to ferrous metals and alloys and it is a cost-effective process.

The various demerits of spray forming are:

1. Spray formed performs has some porosity,

2. Material losses arises from over spray of droplet which do not impact on the substrate and bounce on impacting droplets from the substrate surface, machining losses from out of specification performed.

3. Efficiency of spray forming process is low.

The spray-formed alloys and composites find a wide range of applications in automotive, aerospace, electronic packaging, and electrical industries. Also these are used as bearing materials and tribo-components for tribological

2.2 ALUMINIUM ALLOYS

Aluminium alloys are used in a wide variety of applications because of their attractive properties, Al alloys has high strength, low density, and good wear resistance, low coefficient of thermal expansion. Its low density, which can contribute significantly to the aspect of weight savings in the design and construction of automotive and aerospace components, materials handling equipment, portable tools and even sporting goods. These properties can be improved by the mechanical processing. By these processes mechanical properties such as ultimate strength, yield strength and percentage elongation and microstructure of the Al alloy may be improved.

2.2.1 ALUMINIUM SILICON ALLOYS AND ITS APPLICATION

Aluminium silicon alloys are attractive candidate materials for automotive, aerospace and electronics applications due to their excellent properties, such as low weight, high stiffness, good wear resistance and low coefficient of thermal expansion. These properties can be effectively improved by further increasing the silicon content of the alloys. However, as it is difficult to produce such alloys with high silicon content by means of conventional casting process since coarse primary silicon phase precipitates in the materials and causes reduction of mechanical properties and deterioration of workability. Refinement of primary silicon by adding phosphorus to the molten alloys is insufficient when the silicon content of the alloys is higher than 25 wt. %.

Effective primary silicon refinement can be achieved at increased solidification rates that are facilitated by high under cooling ability. Powder metallurgy seems to be an alternative route for the manufacture of hypereutectic aluminium silicon alloys since the fine powder used is usually highly under cooled and rapidly solidified. Unfortunately,

the need to avoid oxidation and contamination of the powder by complex and costly processing steps hinders the cost-effective application of this process.

Table 2.1 Material characteristics of hypereutectic Al Si alloy spray formed under different thermal condition.

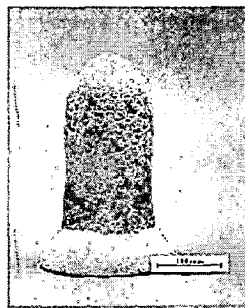
Thermal condition	Hot spray	Cold spray	Optimum spray
Porosity	Round shape	Irregular shape	Few pores
Density	Low	Low	High
Primary silicon	Large particle	Small particle	Small particle
Eutectic phase	Flack	Modified phase	Modified phase
Phase distribution	inhomogeneous	inhomogeneous	homogeneous

2.2.2 SPRAY FORMED BILLET

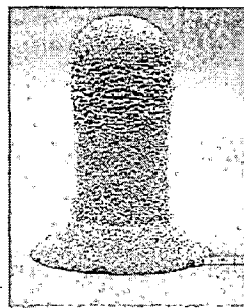
The representative spray formed Al-Si alloy billets are shown in Fig. 2.1. The diameter of the billets is in the range of 130–160mm. The height of the billets is in the range of 400–500mm. The surface of the billets with high content of silicon is generally rough. This indicates relatively slow cooling and solidification of the alloys which contain high amount of enthalpy.

Table 2.2 Main spray parameters for hypereutectic Al Si alloys

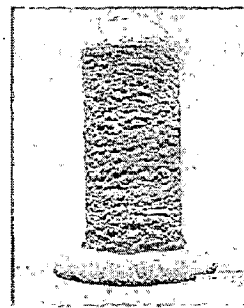
Alloy	Melting temperature	Atomization gas pressure Mpa	Pouring nozzle diameter mm	Spray distance mm	Gas to metal ratio (GMR)	Thermal condition
AlSi18	732	0.4	4.5	440	3.8	Hot
		0.4	4.0		4.0	Hot-
AlSi25	823	0.4	4.0	440	4.0	Hot
		0.5	4.0		7.4	Cold -
AlSi35	953	0.4	4.5	440	5.3	Hot+
		0.6	4.0		7.7	hot



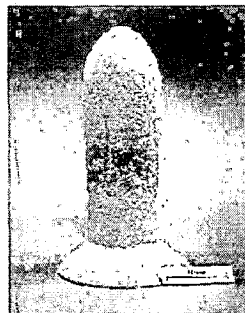
(a) AlSi18,
Hot, GMR=3.8



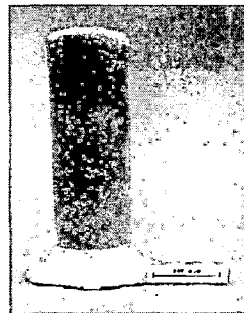
(b) AlSi25,
Hot, GMR=4.0



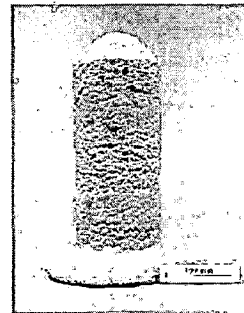
(c) AlSi35,
Hot+, GMR=5.3



(d) AlSi18,
Hot-, GMR=4.0



(e) AlSi25,
Cold-, GMR=7.4



(f) AlSi35,
Hot, GMR=7.7

Fig 2.1 Hypereutectic Al-Si billets prepared under different spray forming conditions

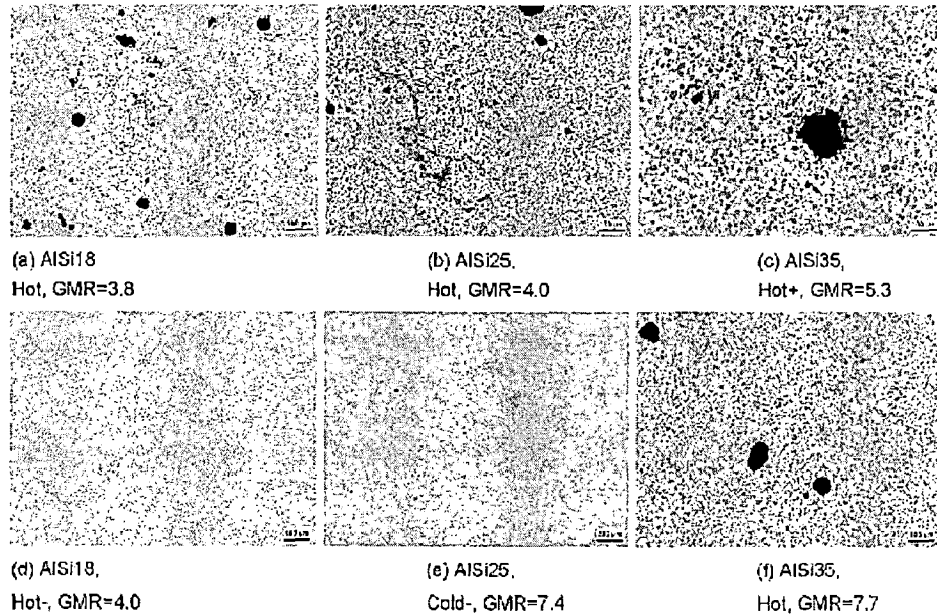


Fig 2.3 Porosity in spray formed hypereutectic Al-Si alloy billets

Porosity is one of the important but undesirable characteristics of spray formed materials. It is proposed that this defect may occur during spray forming as a result of one or a combination of the following mechanisms: interstitial porosity, gas entrapment, precipitation of dissolved gas and solidification shrinkage. The thermal conditions of the impinging droplets influence the top surface region of the deposit and therefore the formation of pores. Interstitial pores are usually found under cold spray condition when the liquid fraction of the droplets is low. The solid particles impact on one another and overlap, leading to interconnected, nonspherical pores if there is insufficient liquid to fill the interparticle or intersplat pores.

Spray forming conditions, especially the cooling and solidification condition, have great influence on the metallurgical quality of the spray formed Al-Si alloys. With the cooling rate of the deposited materials increasing, the size of primary silicon phase in the materials decreases. Interstitial pores appear instead of spherical gas pores if the thermal condition in spray forming is changed from hot condition to cold condition. Eutectic phase in the shape of flake is also modified into short and blunted shape as the deposit solidifies rapidly.

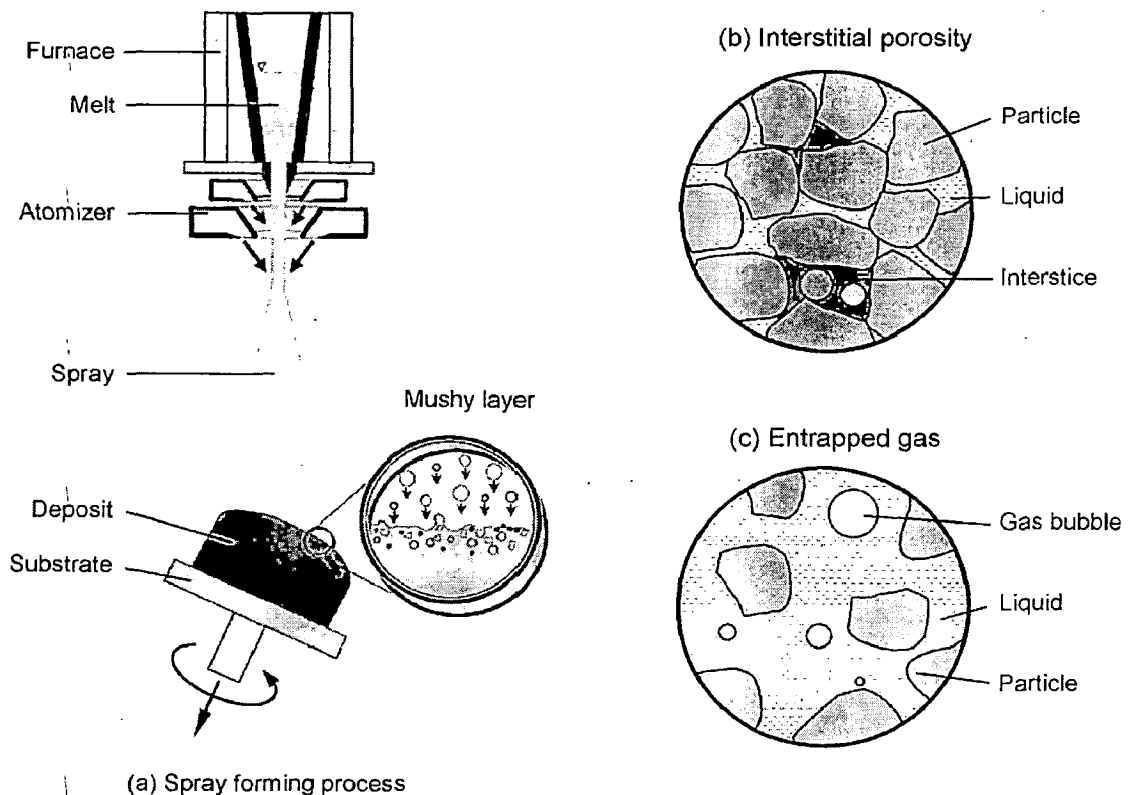


Fig 2.4 Schematic of spray forming and the mechanisms of porosity formation.

Interstitial pores are usually found under cold spray condition when the liquid fraction of the droplets is low. The solid particles impact on one another and overlap, leading to interconnected, nonspherical pores if there is insufficient liquid to fill the interparticle or intersplat pore.

Microstructural features of the perform at different locations from the central and peripheral regions have been observed with a purpose to compare the microstructural variations and also to compare the microstructural characteristics of the spray deposited alloys. Fig. 2.5 shows the microstructural features of spray deposited Al-6.91Si alloy at 30° inclination. The top section (Fig. 2.5a) shows that the porosity (size 20–30 μ m) is very less on top surface. The middle section (Fig. 2.5b) shows more porosity than that of top section and bottom section (Fig. 2.5c) than that of middle section of the deposit. Similarly, the amount of porosity of the peripheral region (Fig. 2.5d) of the deposit was observed to be higher than those of central region.

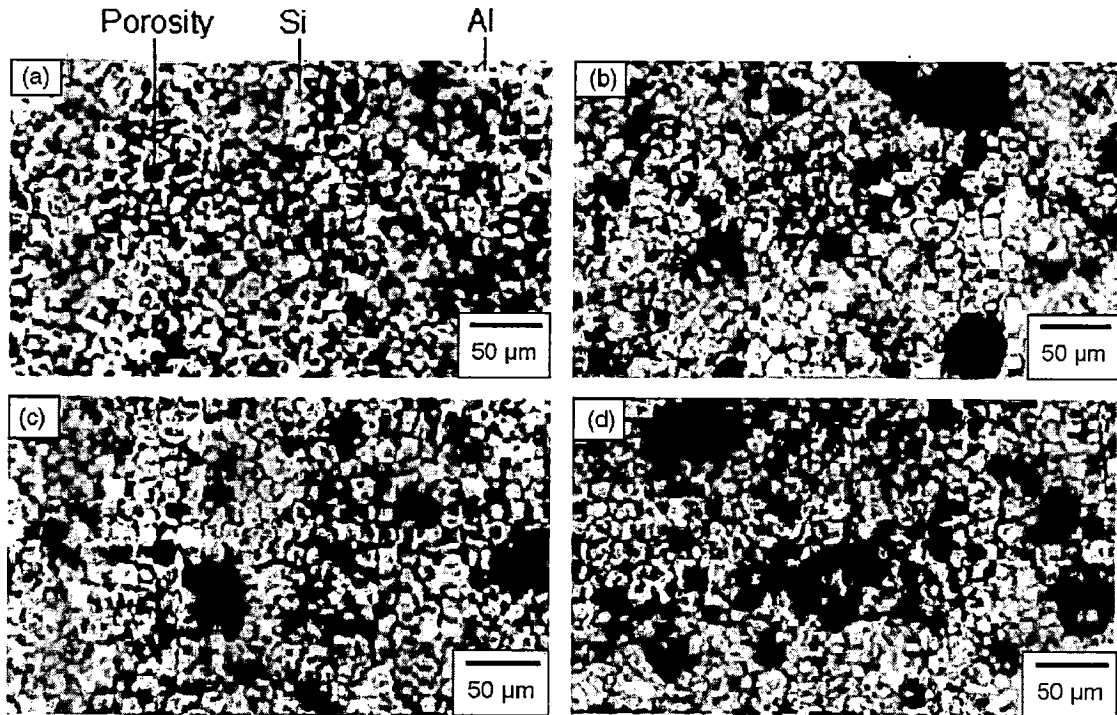


Fig. 2.5 Micrographs of spray deposited Al-6.91Si alloy at four different sections (a) top, (b) middle, (c) bottom and (d) Peripheral.

The atomization process plays an essential role for processes like spray forming as it primarily influences the shape of the sprayed deposit as well as its material properties. In the atomization stage, the mass flux distribution and enthalpy density distribution in the spray are determined, resulting from the different sizes of droplets and particles. In comparison to conventional liquid atomization processes (e.g. atomization of water based liquids), atomization of metal melts in spray forming is more complicated due to the high temperature of the hot melt and the high temperature gradients between the melt and the cold atomization gas. In addition the material properties of both phases change over a wide range due to temperature variations. The purpose of the investigation is to understand and describe the main driving mechanisms of the atomization process as a base for modelling of the overall disintegration process and to derive possibilities of direct influencing the resulting spray properties from the atomization process.

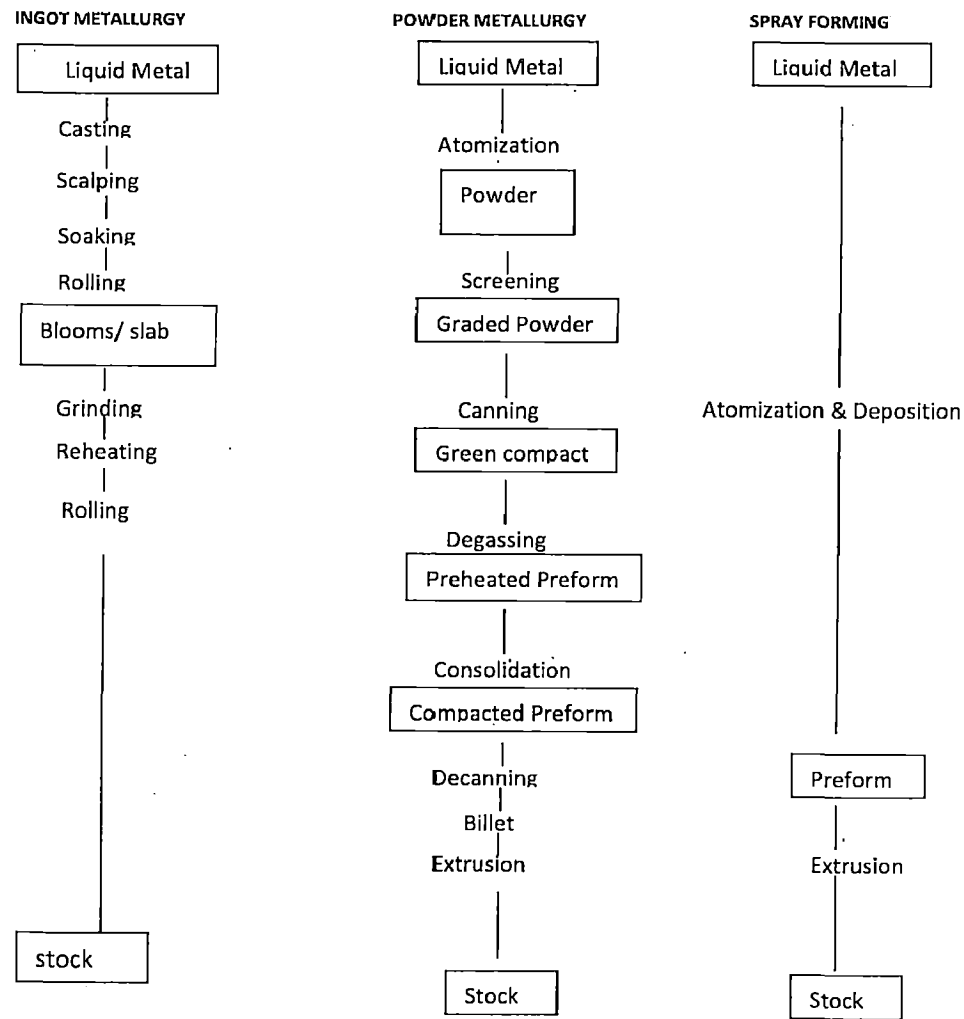


Fig 2.6 reduction of process steps for spray formed reformed in comparison with powder metallurgy

2.3 MECHANICAL PROCESSING OF Al-Si ALLOYS

Fig. 2.7 shows the microstructure of the extruded (A) and the extruded/ heat-treated (B) 380 alloy. The microstructures observed in both cases present distributed silicon particles in an Al matrix and intermetallics phases containing iron and copper (brighter phases). The microstructure of the spray formed materials show significant differences to conventionally cast counterparts. It is possible to recognize an equiaxial, refined Al matrix and near-uniform distributed silicon particles having an estimated maximal length of 15 μm . The equiaxial appearance of the Al phase is a remarkable feature of the

observed microstructures and is ascribed to the extensive fragmentation and coarsening of solid phases during the build-up of the deposit.

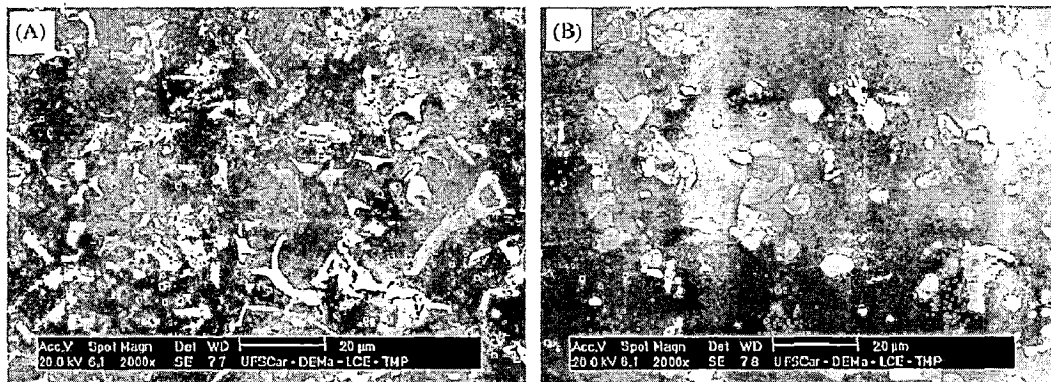


Fig 2.7 SEM images of the extruded (A) and extruded + heat treated 380 alloy (B).

The remarkable difference between the “as-sprayed deposit and the extruded one is that the extrusion process reduced considerable the porosity volume levels from approximately 4–0.5%. No significant difference was observed in the other microstructural features such as the aspect ratio of the silicon particles or distribution/size of the intermetallics, a result that can be attributed to the low reduction rate (5:1) applied during the extrusion. Regarding microstructural changes the solution heat treatment carried out at 783K played a more important role and reduced significantly the aspect ratio of the silicon particles (compare Fig. 2.7A and B), and, as well as the quantity and morphology of the intermetallics (brighter phases in Fig. 2.7A and 1B). The silicon phase was not associated with an eutectic structure in this work, but rather as isolated particles similar to primary silicon in hypereutectic alloys. This fact was already observed in a previous work as well as documented by other investigators working on spray forming of eutectic and hypereutectic aluminium–silicon alloys . In order to exply the particulate morphology of silicon we suppose that the impacting droplets break up the growing eutectic, including the silicon plates, as they do with the Al dendrites. In such way the broken silicon plates have their aspect ratio strongly reduced resulting in the particulate morphology (Fig. 2.7A). Further, these particles lost their still remained sharp edges, if any, by the influence of the heat treatment (Fig. 2.7B), which led to a significant increase in the elongation to fracture in tensile tests,. Heat-treated materials showed higher values for yield stress, YS, and ultimate tensile stress, UTS, than the material that was just extruded. This behavior was accompanied by

an increase in elongation as well. The gain in YS and UTS were expected with the heat treatment. However, such treatment should lead to a decrease in the elongation to fracture as is well known for precipitation hardening alloys.

alloy after hot pressing, which is different from that of as-deposited preforms. There aren't porosities in the pressed alloy. Moreover, the primary silicon and aluminium matrix realign. The primary silicon congregates in microzone under the stress, and aluminium matrix flows into the porosities and connects in hardness.

2.3.1 ROLLING OF Al-Si ALLOYS

AZ91 magnesium alloy was prepared by spray forming. The spray-deposited alloy was subsequently hot-rolled with a 80% reduction at 350 °C. The microstructural features of the as-spray-deposited and hot-rolled alloy were examined by optical microscopy, scanning electron microscopy and X-ray diffraction. The results show that the spray-formed AZ91 magnesium alloy has, compared with the as-cast ingot, a finer microstructure with less intermetallic phase Mg₁₇Al₁₂ dispersed in the matrix due to fast cooling and solidification rates of spray forming process, and, therefore showing excellent workability. It can be hot-rolled with nearly 20% reduction for one pass at lower temperatures (330-360°C), and the total reduction can reach 50% prior to annealing. After proper thermo-mechanical treatment, the spray-formed AZ91 magnesium alloy exhibits outstanding mechanical properties. Hot rolling techniques have extensive applications in wrought magnesium alloys, therefore, in the present work, AZ91 magnesium alloy was prepared by spray forming, the feasibility of hot rolling for this alloy at lower temperatures (330-360°C) was investigated, and the hot rolling characteristics of spray-formed AZ91 alloy were discussed. The microstructures of the as-sprayed AZ91 magnesium alloy are shown in Fig. 1. Compared with the as-cast ingot, the spray-formed AZ91 alloy has a finer microstructure with less intermetallic phase Mg₁₇Al₁₂ dispersed in the matrix. The average grain size is about 20 μm. This feature is invariably present in the samples from different regions. Fig. 2 shows XRD patterns of as-cast and as-deposited AZ91 Mg alloy. The diffraction pattern of the spray-formed AZ91 magnesium alloy.

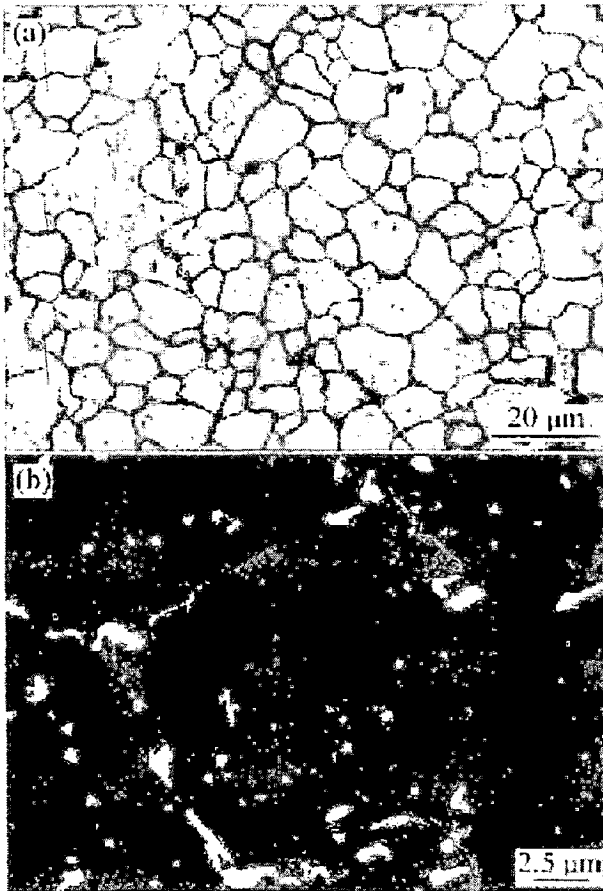


Fig 2.8 Microstructures of as-deposited AZ91 magnesium Alloy (a) Optical microstructure; (b) SEM

CHAPTER-3

FORMULATION OF PROBLEM

After reviewing the literature critically and with the importance of Al-Si alloys, the scope of the problem and objectives of the present study have been formulated which are as follows.

- Preparation of Al –Si alloy using spray forming technique.
- To study of the microstructure of spray formed Al- Si alloy at the different Percentage reduction of rolling.
- To study of the mechanical properties of spray formed Al- Si alloy at the different Percentage reduction of rolling.
- Wear behavior of Al-Si alloys before and after the rolling.
- To study of the density and porosity of Al – Si alloys at different percentage reduction of rolling.

CHAPTER-4

EXPERIMENTAL SETUP AND PROCEDURE

In this chapter the experimental details used for the study has been summarized. It includes the alloy chemical composition, mechanical testing, metallographic examination, and wear testing.

4.1 SPRAY FORMING SETUP:

The schematic of spray forming set-up is shown in Fig. 4.1. The photograph of spray forming set-up is shown in Fig. 4.2. The atomizer was placed above the cooling chamber and the resistance heating furnace is placed above the atomizer. The alloy was allowed to melt into a crucible. The melt flow tube connected to crucible passes through the furnace to cooling chamber via atomizer. The atomized droplets were collected over the copper substrate.

About 1 kg Al-Si alloy of different composition of %Si was superheated to 200 K above the melting temperature in the crucible to carry out different runs. Atomization of melt results in a spray of wide range of micron size droplets. These droplets were allowed to deposit over the copper substrate. Preforms were taken out of the substrate after deposition.

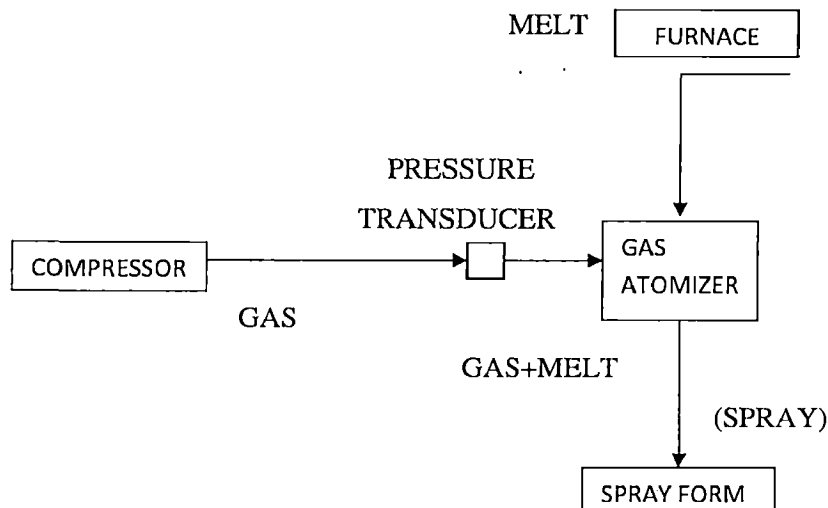
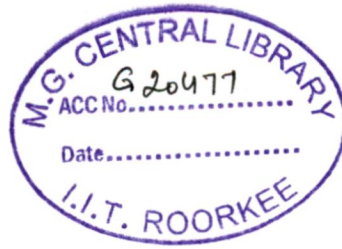


Fig 4.1 SCHEMATIC OF SPRAY FORMING SET-UP



4.2 MICROSTRUCTURE

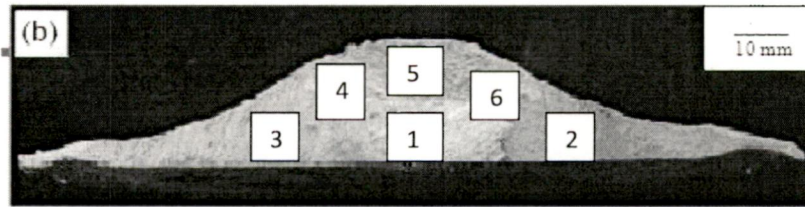


Fig 4.2 cross section preform

Samples from the different parts of spray formed deposits were cut down for its micro structural study as shown in fig 4.2 at locations 1, 2 and 3. These samples were polished using standard metallographic technique of polishing with an emery paper of 1/0, 2/0, 3/0 and 4/0 specification and then followed by wheel cloth polishing using an emulsion of alumina powder particles suspended in water. Afterwards these samples were polished by kerosene oil and brasso. Then these samples were etched with Keller's reagent (composition: 1% vol. HF, 1.5% vol HCl, 2.5% vol. HNO₃ and remaining water). The microstructures of samples were examined under Letiz optical microscope and JEOL JXA840A scanning electron microscope.

4.3 HARDNESS TESTING

Vickers hardness (HV_s) of all the Al-Si alloys was measured using brinell cum Vickers hardness tester of model HPO 250 at 5 Kg load for Al-Si-Pb alloys. The samples were cut down from different radial locations of preform and polished by emery paper and cloth using alumina suspended in water. As a matter of routine practice, indentation at least 20 different locations were taken and the resulting hardness values averaged to obtained the mean hardness value.

4.5 WEAR TESTING:



Fig 4.3 Pin on disk wear testing set up

Wear experiments were conducted on a pin-on-disc machine. The machine contains a ground hardened EN-24 steel disc (0.12 m in diameter) of surface roughness 0.4-0.5 μm centre line average with a Rockwell hardness of 57 HRC. The steel disc was kept rotating at a constant speed (2 ms^{-1}) in the present investigation. Experiments were carried out dry at a relative humidity of approximately 30% at a temperature of 309 K. A load cell was fixed in contact with the holder arm. The load cell is connected to a simulator to facilitate measurement of the frictional force of the specimen near the interface due to frictional. It may thus be used as a measure of the frictional resistance between mating surfaces.

4.51 TESTING METHOD

Procedure

1. The four specimens were prepared for the wear test with the of belt grinder and polished on emery papers 1/02/0. Then the original length, diameter, and weights are noted down.
2. The specimen is mounted in the holder and it is kept on the rotating disk. The disc is cleaned properly before test conducted.
3. Coefficient of friction is noted down after every 30 second.
4. Weight loss is measured after every 20 minutes at different load.

4.6 MEASUREMENT OF POROSITY AND DENSITY:

Density of the Al-Si alloy specimen was measured using Archimedes principle. Distilled water was employed for these measurements. Keroy single pan balance capable of weighing accurately up to 0.00001 gm was employed for these estimations. In each case, at least four readings were taken and 2 readings closest to each other were taken to be the correct value. Specimens for these measurements were fabricated from the central region of the preform. Density & porosity were calculated as follows



Fig 4.4 Density measurement set up

If the mass of sample measured in air is = m_a , g

The mass of sample measured in water = m_w , g

Density of water = ρ_w , g/cm³

The theoretical mass or density of the sample per unit volume = m_t , g/cm³

According to Archimedes principle, density = $m_a \rho_w / (m_a - m_w)$

Porosity = $[m_t - \{m_a \rho_w / (m_a - m_w)\}] * 100 / m_t$

5.2 Spray formed characterization

Preforms produced under different composition are characterized by the properties reported below:

Microstructural Features

Samples are cut from the preforms as shown in fig. 5.1 for observation were prepared following standard metallographic procedures.

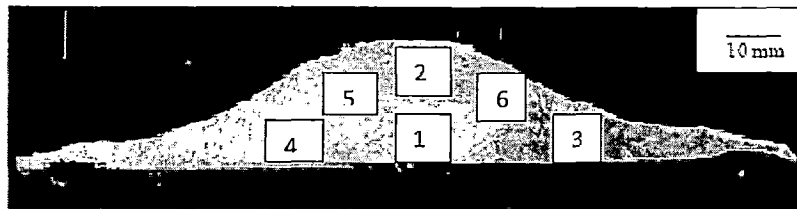


Fig 5.1 cross section of spray form showing location from where samples are cut

The spray forming process exhibit considerable microstructural refinement in Al-Si and Al-18Si alloys . The size of the primary Si phase with a particulate morphology varies from 3 to 7 μm in the spray deposit of Al-Si alloy against their size of 100-130 μm in the as cast alloy. There is no segregation of second phase in the binary alloys. Generally equiaxed grains are found in the spray formed preform of Al-Si alloys.

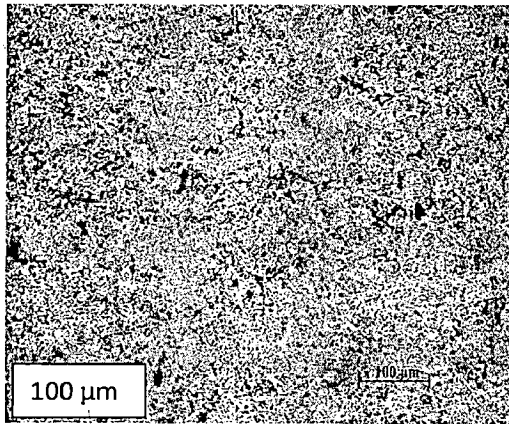
Microstructural features of the preform at different locations from the central and peripheral regions have been observed with a purpose to compare the microstructural variations andalso to compare the microstructural characteristics of the spray deposited alloys.

Fig. 5.2 shows the microstructural features of spray deposited Al-Si alloy. The top section (Fig. 5.2a) shows that the porosity is very less on top surface. Microstructure are taken at the distance between point 10 mm. The middle section (Fig. 2b) and (Fig. 5.2c) shows more porosity than that of top section and bottom section (Fig. 5.2d) than that of

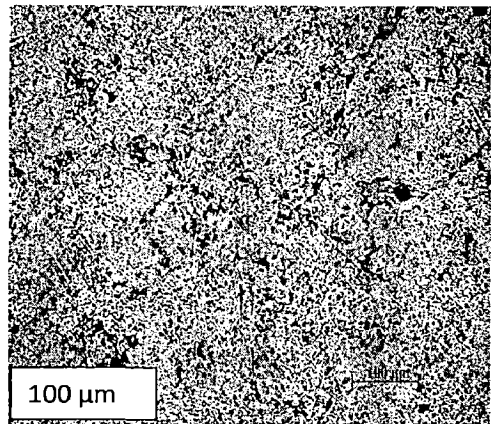
middle section of the deposit. Similarly, the amount of porosity of the peripheral region of the deposit was observed to be higher than those of central region. a,b,c is taken in the radial direction. porosity is less in the 5.2 d, e, f as compare to the fig 5.2 a,b, c .

Fig 5.3 shows the change in the microstructure .when the 5 % rolling is done the reduce in the porosity and fine grains are produced. Which is in the figure 5.3 (a,c,e) while in figure 5.3 b,d,f taken microstructure in the radial direction. Radial direction has less porosity because less gas entrapment in the deposit . at the radial speed of inert gas is less as compare to the central portion of the perform . shows the reduced the porosity .

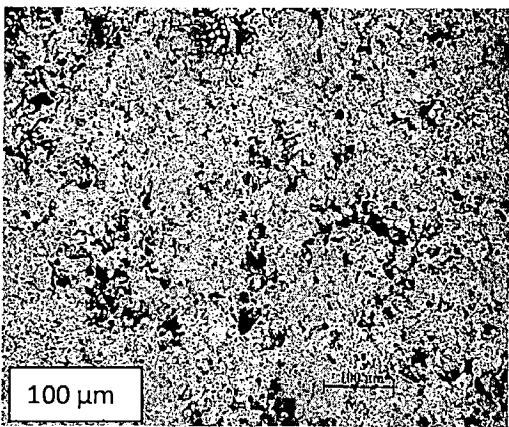
Figures 5.4 show the grain elongation and fine grain size . more grains elongation in the direction of rolling because at this stage of rolling metal flows in the direction of rolling. this is due to the At the 15 percent of reduction in thickness more grain elongation take place in the direction of rolling. The primary silicon congregates in microzone under the stress, and aluminium matrix flows into the porosities and connects in harness.



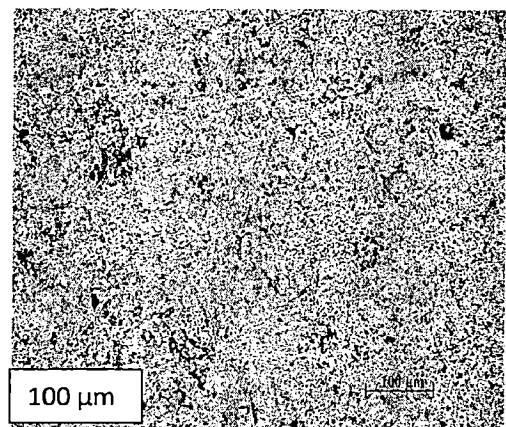
a



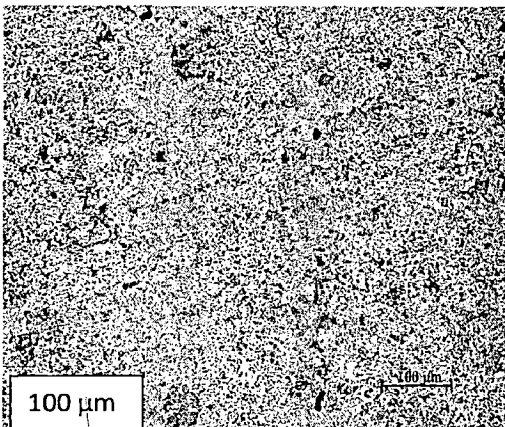
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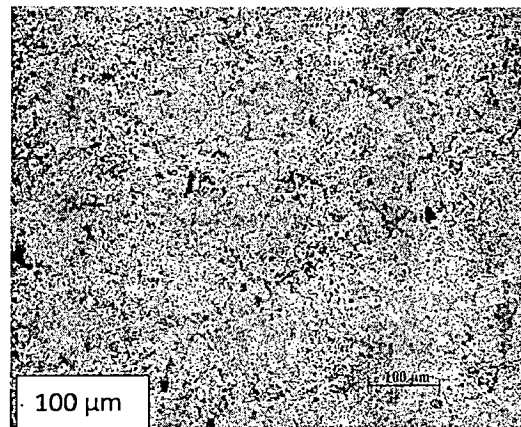
c



d

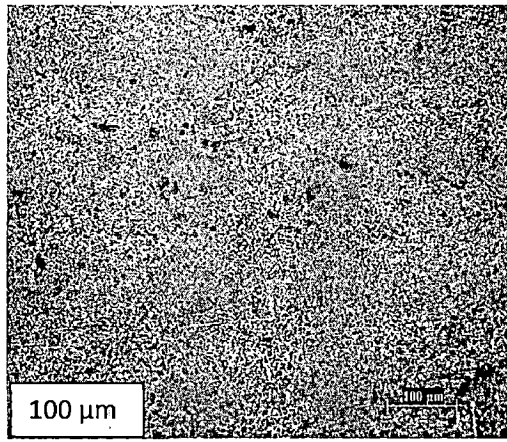


e

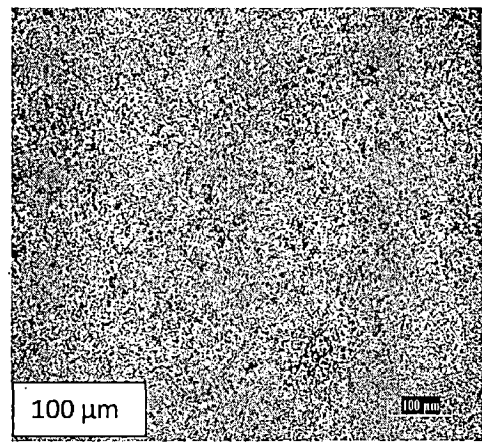


f

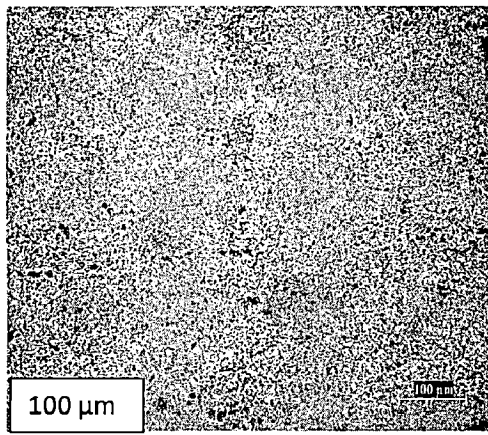
Fig 5.2 Microstructure of spray deposited Al-Si alloys without rolling (a) 1 (b) 2(c) 3 (d) 4(e) 5(f) 6, Location shown in figure



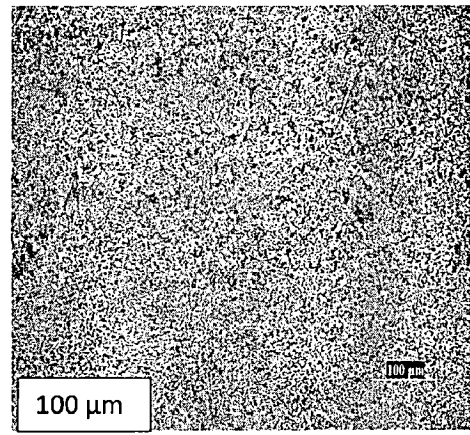
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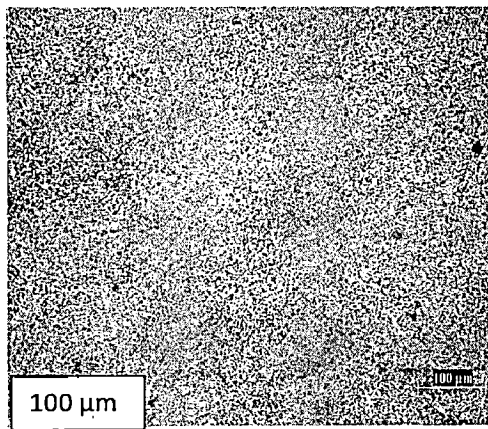
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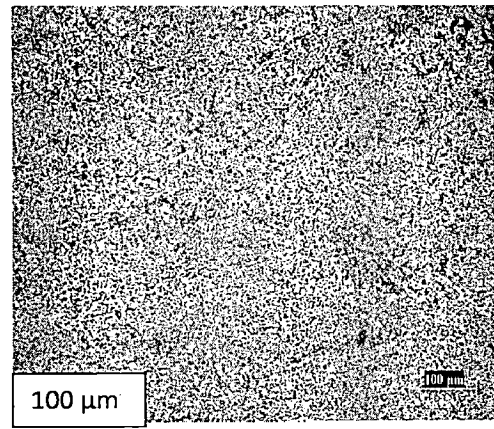
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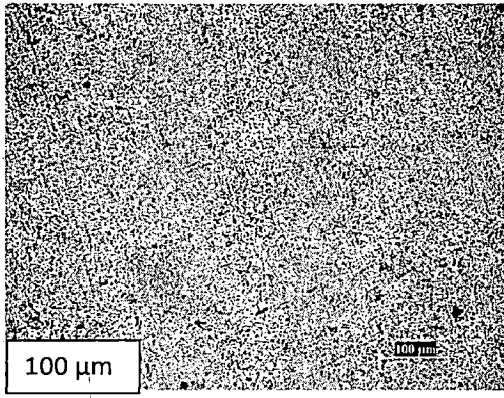
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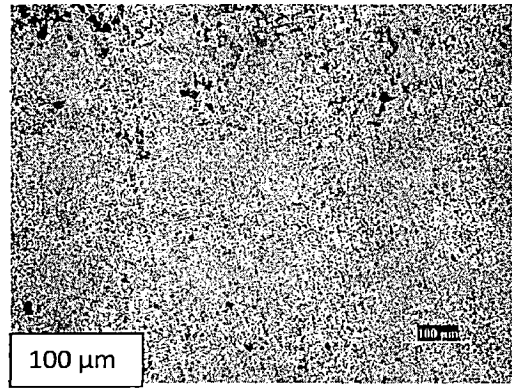
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Fig 5.3 Microstructure of spray deposited Al-Si alloys with 5 %rolling (a) 1 (b) 2(c) 3 (d) 4(e)

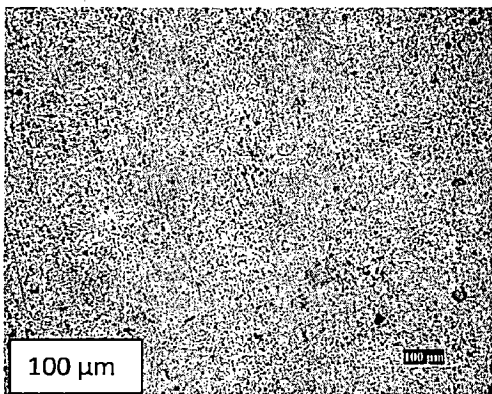
5(f) 6, Location shown in figure



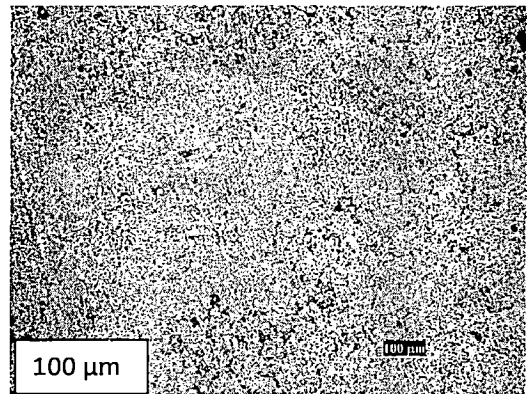
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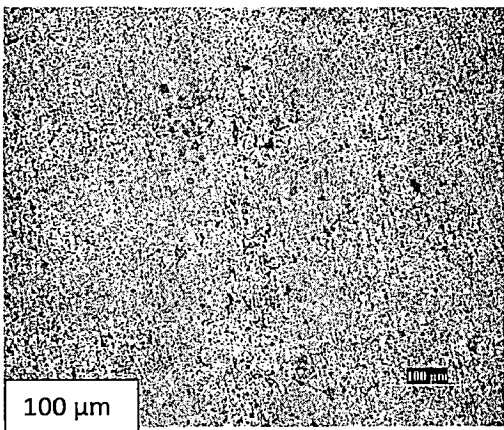
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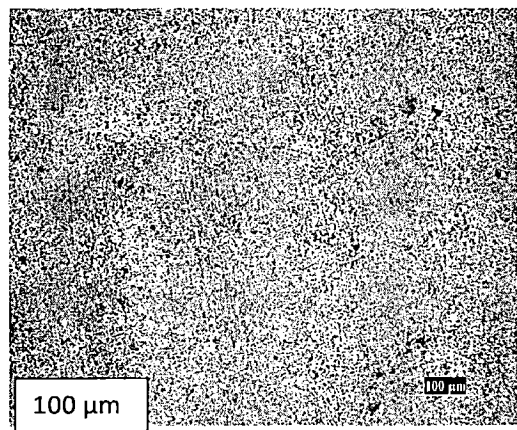
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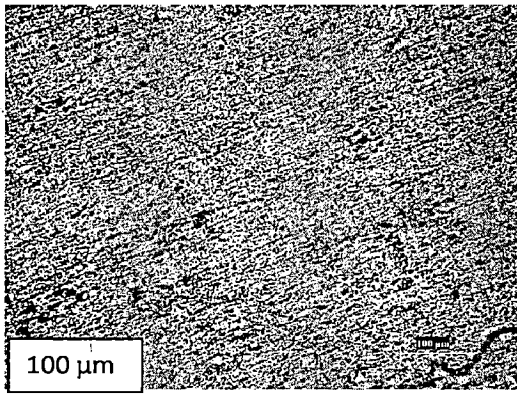
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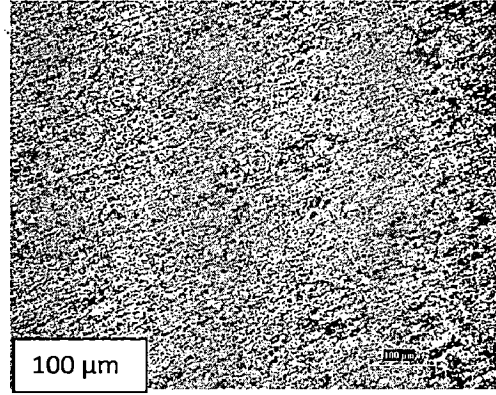
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Fig 5.4 Microstructure of spray deposited Al-Si alloys with 10 % rolling (a) 1 (b) 2(c) 3 (d) 4(e)

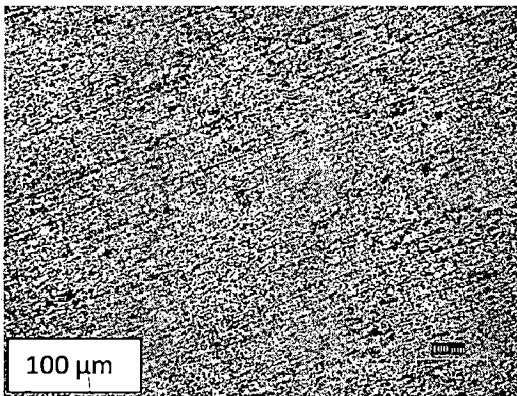
5(f) 6, Location shown in figure



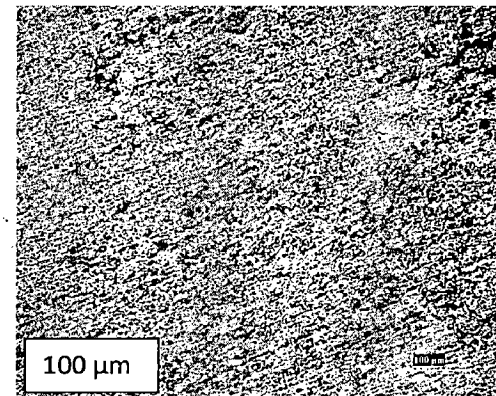
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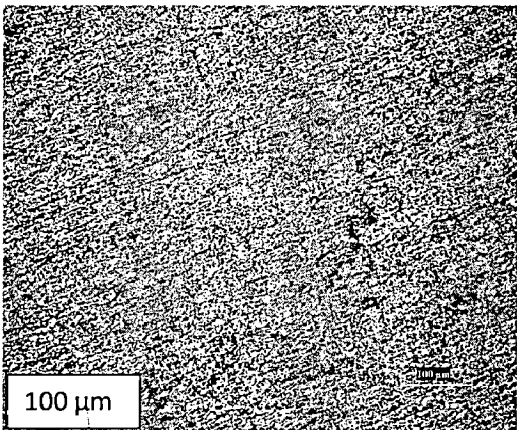
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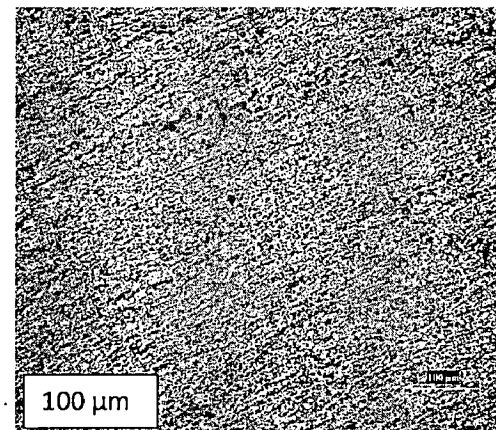
c



d



e



f

Fig 5.5 Microstructure of spray deposited Al-Si alloys with 15 % rolling (a) 1 (b) 2(c) 3 (d) 4(e)

5(f) 6, Location shown in figure

5.2 HARDNESS

One half of the sample strip was analyzed under Vickers hardness machine. Hardness value at different location were taken as shown in fig 5.10. Table 5.1 to 5.4 gives the value of hardness at different points shown in figure at the different percentage of reduction of rolling. Hardness values were taken at 20 points. Load of 5 kg was applied while measuring the Vickers hardness

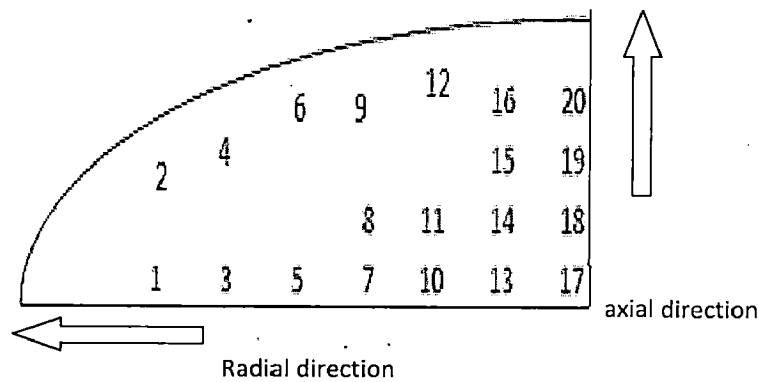


Fig 5.6 Hardness value taken at different points

Hardness vary in the radial as well as in the thickness direction of the perform because of the cooling rate vary. Cooling is fast in the radial or surface direction so at the edge fine grains are form which lead to increase the hardness. As we can see in table 5.1 to 5.4 , hardness value of preform vary in range shown in tables. maximum hardness taken from all of the table which is the 0 ,5 ,10 and 15 percentage of rolling .increasing in the hardness (42.7, 49.9 52.3 and 54.4)s with the reduction percentage of rolling .this variation in hardness due to collapsing and fragmentation of pores .area of pores is fill by the Al-Si deformation metal is flow in the vacant space in axial direction when initial stage of rolling 5% . after that thickness reduction more very small amount of porosity is present and flow of metal is stop in the pores . so increase in hardness which is show in figures of different percentage of rolling.

Table 5.1 Vickers hardness under load of 5 kg (without rolling)

Point location from figure	Average diagonal	Hardness (VHN)
1	0.467	42.5
2	0.478	40.6
3	0.466	42.7
4	0.484	39.6
5	0.501	37.0
6	0.505	36.4
7	0.497	37.5
8	0.503	36.7
9	0.513	35.2
10	0.507	36.1
11	0.505	36.4
12	0.557	29.87
13	0.513	35.2
14	0.552	30.53
15	0.574	28.13
16	0.557	28.03
17	0.557	29.87
18	0.618	24.25
19	0.581	27.46
20	0.606	25.18

As we can see that from the above table and graph that as we move along the radial direction hardness of preformed increased or little variation. This variation in hardness due to the porosity present in the preformed and also in the case of at different percentage of reduction of rolling. Which are given in the table 5.2 to 5.4 .same is in the case when we move along the axial direction. These hardness values are plotted in various graphs which will help us in understanding the profile along the section.

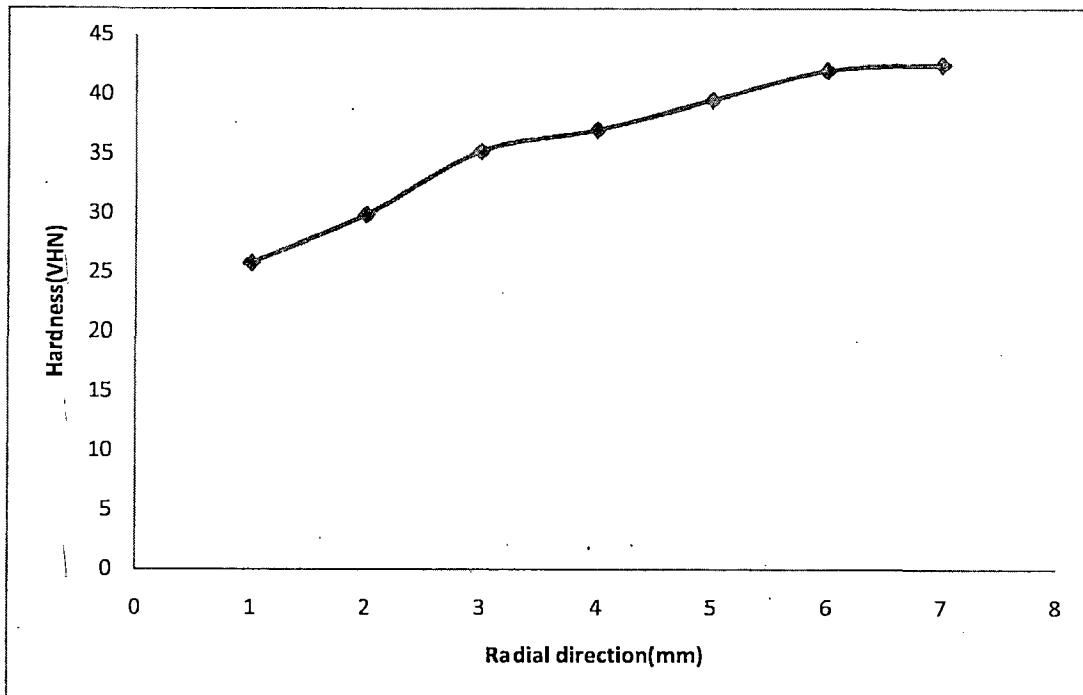


Fig 5.7 Hardness variation along radial direction

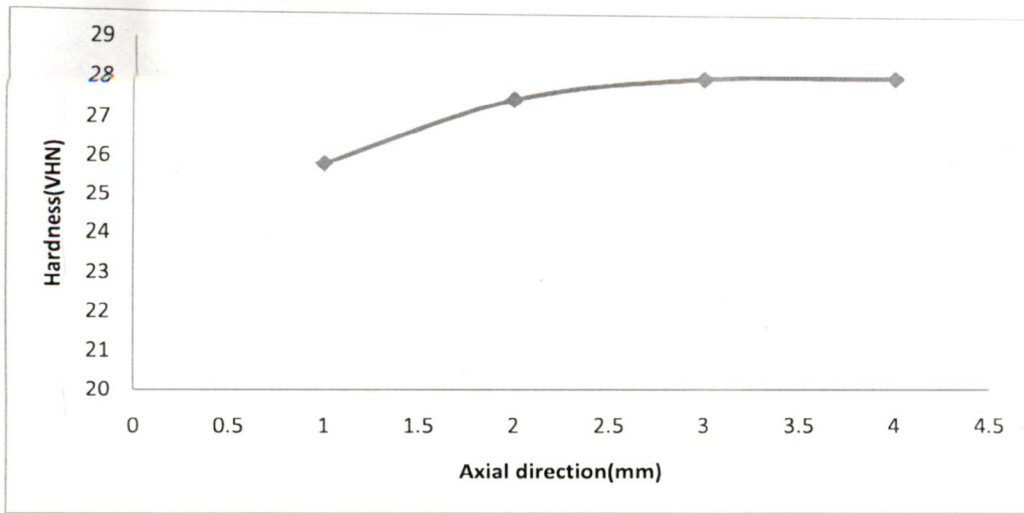


Fig 5.8 Hardness variation along axial direction

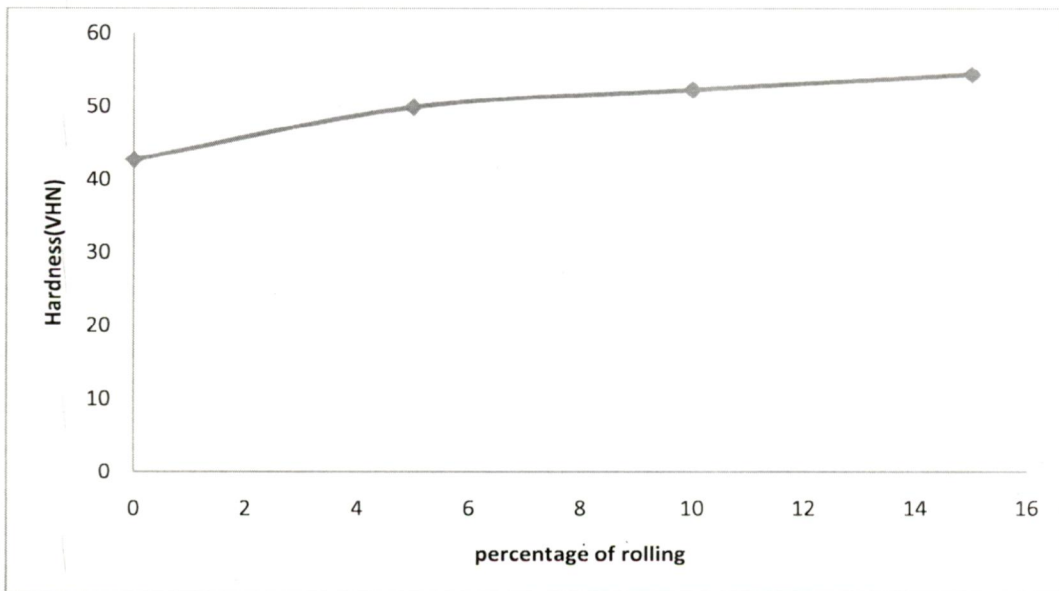


Fig 5.9 variation in hardness with the percentage of rolling

Table 5.2 Vickers hardness under load of 5 kg (5 percentage rolling)

Point of location	Average diagonal	Hardness (VHN)
1	0.431	49.9
2	0.436	46.6
3	0.432	49.7
4	0.448	46.2
5	0.436	48.8
6	0.450	45.8
7	0.449	46.0
8	0.453	45.2
9	0.474	41.3
10	0.467	42.5
11	0.452	45.4
12	0.453	45.2
13	0.454	45.0
14	0.45	45.8
15	0.462	43.6
16	0.464	43.1
17	0.476	40.9
18	0.491	38.5
19	0.474	39.6
20	0.50	37.1

Table 5.3 Vickers hardness under load of 5 kg (10 percentage rolling)

Point o location	Average diagonal	Hardness (VHN)
1	0.421	52.3
2	0.420	52.6
3	0.419	52.8
4	0.426	51.1
5	0.422	52.1
6	0.424	51.6
7	0.426	51.1
8	0.425	51.3
9	0.429	50.4
10	0.461	43.6
11	0.430	50.2
12	0.437	48.6
13	0.441	47.7
14	0.442	47.5
15	0.428	50.6
16	0.440	47.9
17	0.461	43.6
18	0.430	50.2
19	0.452	45.4
20	0.479	40.4

Table 5.4 Vickers hardness under load of 5 kg (15 percentage rolling)

Point o location	Average diagonal	Hardness(VHN)
1	0.413	54.4
2	0.414	54.1
3	0.41.8	53.1
4	0.417	53.3
5	0.417	53.3
6	0.416	53.6
7	0.422	52.1
8	0.423	51.8
9	0.426	51.6
10	0.429	50.4
11	0.431	49.1
12	0.432	49.7
13	0.435	49
14	0.437	48.6
15	0.444	47.0
16	0.452	45.4
17	0.464	43.1
18	0.481	40.1
19	0.464	43.1
20	0.486	39.3

5.3 WEAR PROPERTIES

The wear rate of spray formed alloys was observed to increase with an increase in applied load. It is observed that, when the applied load is increased and crosses the transition point, a significant increase in wear rate takes place. Subsequently, this value remained constant in a wide range of applied load. A wide difference was noticeable in the value of coefficient of friction of these alloys.

Wear Test Conditions

Sliding distance (m) 2400
Applied load (N) 10, 20, 25, 30, 35
Sliding time (min) 20
Sliding speed (m/s) 2

Table 5.5 Variation in wear rate of spray formed alloys as a function of applied load

Load(N)	Wear Rate
10	34.50
20	49.35
25	54.64
30	64.45

Fig 5.10 shows that coefficient of friction is almost constant when plotted against the time. Time is taken in the second. So we can say that coefficient of friction does not depend on time. There is little variation which may occur due to the change in temperature and environmental condition.

Figure 5.12 shows the variation in wear rate as the function of load. Wear rate is increases as the load is increases. Load is increases then friction between disc and samples increases, so weight loss increases as the load increases.

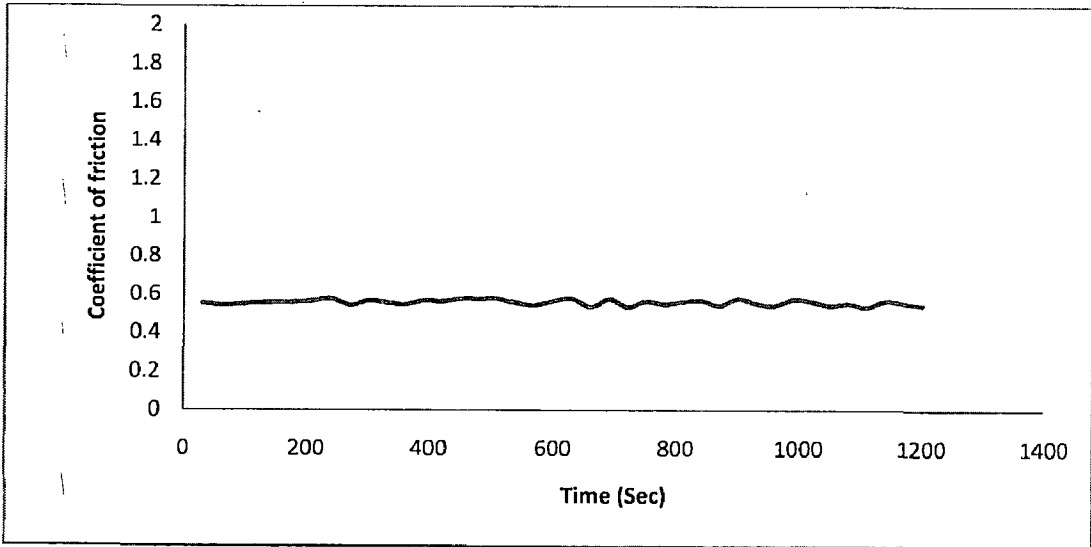


Fig 5.10 variation in the coefficient of friction as the function of time

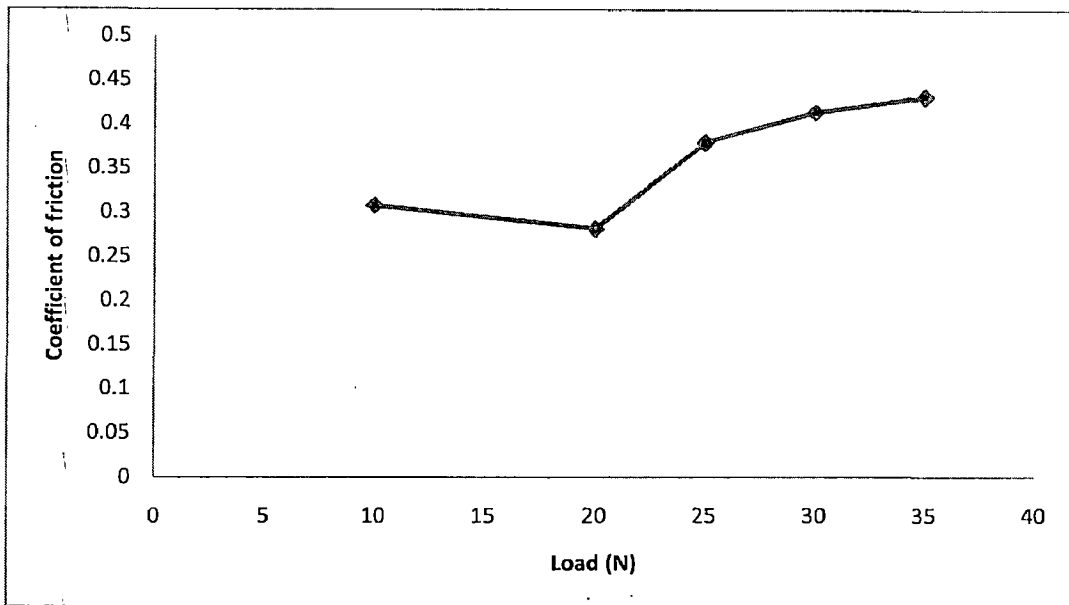


Fig 5.11 variation in the coefficient of friction as the function of applied load

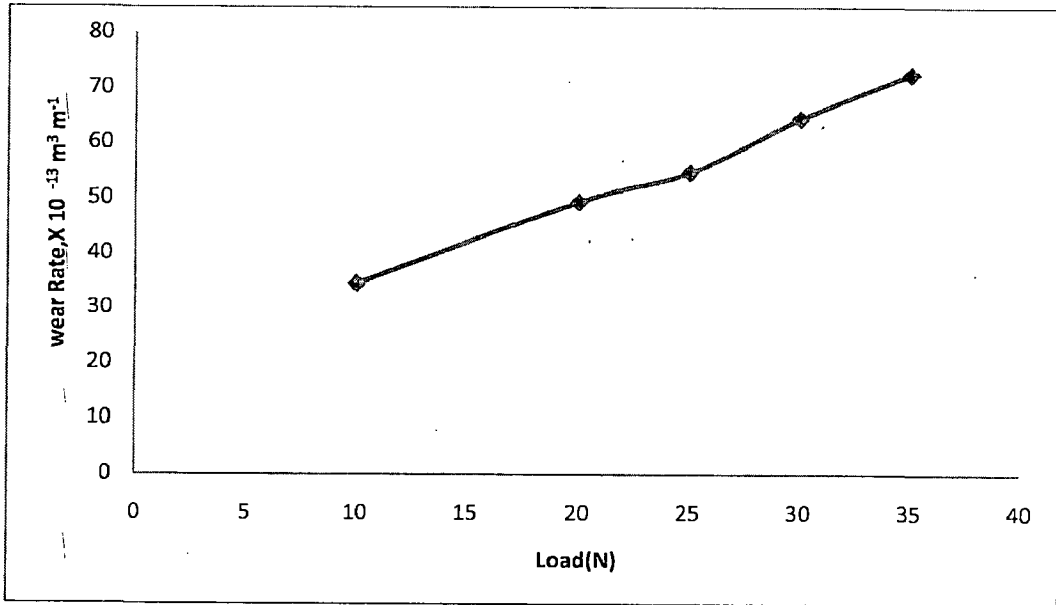


Fig 5.12 variation in the wear rate as the function of applied load

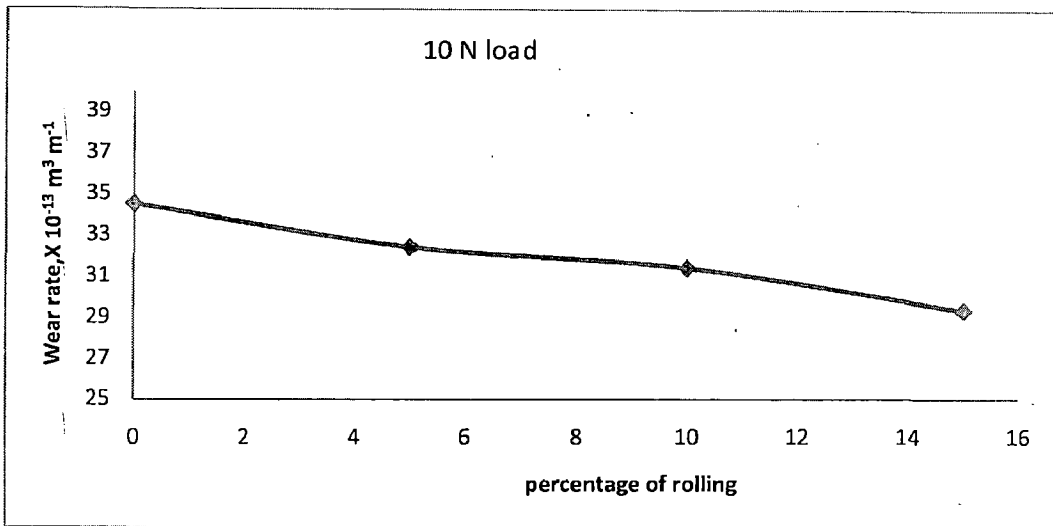


Fig 5.12 variation in the wear rate as the function of rolling

In summary, the results of the present investigation indicate that a modification in spray forming technique for processing of liquid immiscible alloys facilitates a uniform dispersion of Pb in the Al-phase. An optimization of the process variables in this process generates materials with considerably improved wear characteristics.

5.4 DENSITY AND POROSITY

Porosity is one of the important but undesirable characteristics of spray formed materials. this defect may occur during spray forming as a result of one or a combination of the following mechanisms: interstitial porosity, gas entrapment, precipitation of dissolved gas and solidification shrinkage. The thermal conditions of the impinging droplets influence the top surface region of the deposit and therefore the formation of pores. Interstitial pores are usually found under cold spray condition when the liquid fraction of the droplets is low. The solid particles impact on one another and overlap, leading to interconnected, non-spherical pores if there is insufficient liquid to fill the antiparticle or intersplat pores. The density decreases with the increase in percentage of Si in Al-Si alloys.

Table 5. 6 Variation in density of spray formed alloys as a function of rolling

Percentage of rolling	Density(gram / cm ³)
0	2.25
5	2.5936
10	3.036
15	3.1354

Table 5. 7 Variation in porosity of spray formed alloys as a function of rolling

Percentage of rolling	Porosity(percentage)
0	8.1
5	6.2
10	3.8
15	2.8

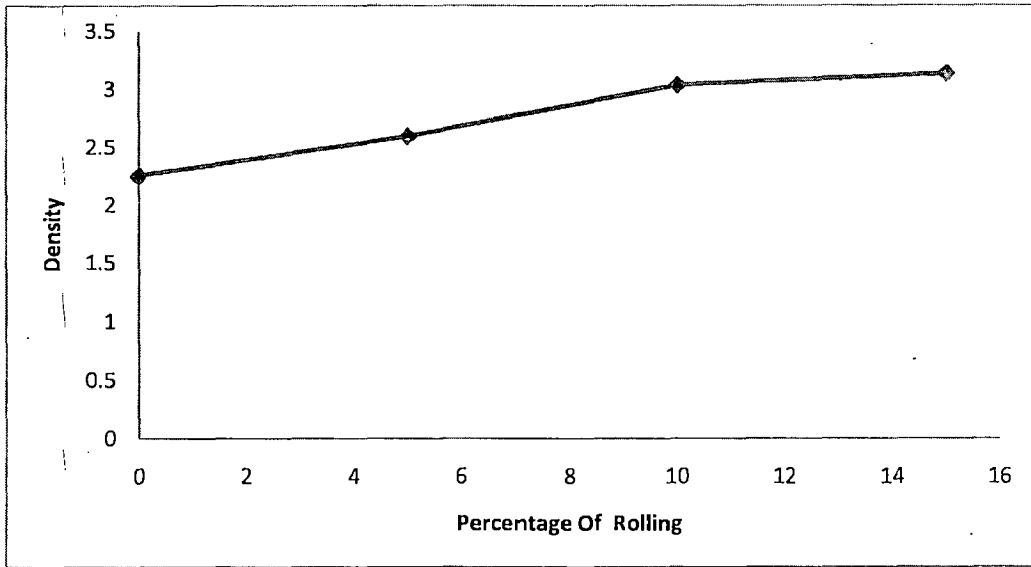


Fig 5.13 variation in density as the function of percentage of reduction rolling

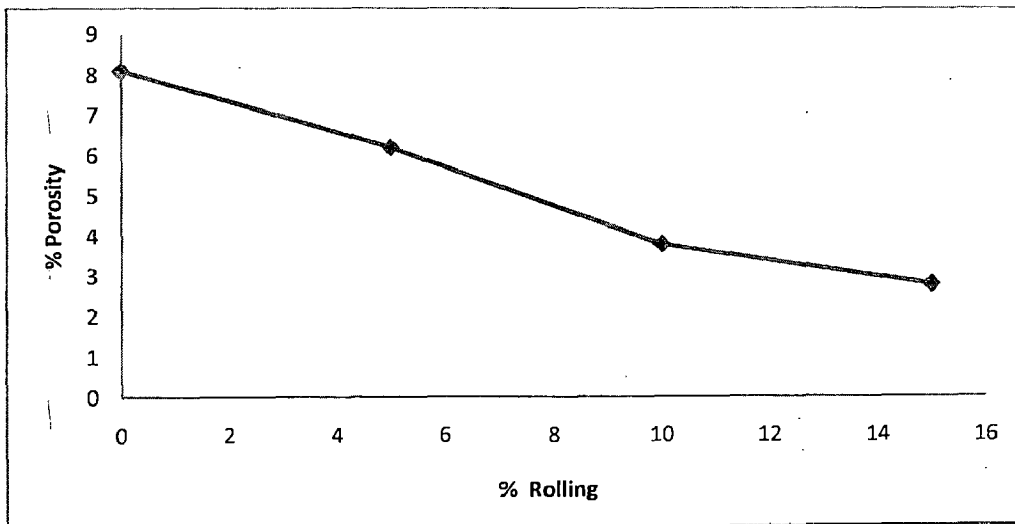


Fig 5.14 variation in porosity as the function of percentage of reduction rolling

6.1 MICROSTRUCTURE

There is grain refinement in the spray casting as compared to I/M cast Al-Si alloys. Grain refinement in the spray casting over that of I/M cast takes place due to high value of heat transfer coefficient and also due to nucleation sites created by the dendrites fragmentation. Both of these phenomena can be explained as follows. The grain refinement due to heat transfer could be understood in the light of the mechanisms involved during spray deposition. A wide size range of droplets created during atomization is propelled to the substrate under the effect of high speed gas jet. Droplets lose their heat to the atomization gas by convective heat transfer. The convective heat transfer takes place rapidly due to high speed gas jet. These droplets therefore experience a cooling rate in rapid solidification regime. Heat transfer coefficient is a function of gas speed therefore it will vary in both directions axial as well as radial in gas field. The microstructure of the perform should also vary with radial as well as axial distance. Axial distance is kept optimum to get a coherent phase of the preform. In radial direction the microstructure should be coarse grained at the periphery, as compared to central region, due to low speed of gas at periphery. But it was not the case, the periphery was found to have fine grained microstructure. The reason for it could be higher rate of conductive heat transfer ($Q \propto 1/x$) at periphery due to its low thickness as compared to that of central region. Grain refinement due to dendritic fragmentation could be understood as follows. As the thickness of the spray deposit increases a semi-molten liquid pool is created on the deposition surface with a turbulent fluid flow condition due to the high speed gas stream. This effect induces shearing action and fragmentation of dendrites into tiny particles. The tiny particles act as a nucleation sites and give rise to grain refinement.

At the initial 5 % percentage of rolling the little grain elongation take place in the radial but more in the thickness direction because of more metal flow in that direction due to the less stress produce in the thickness direction. however the grain are fine because the collapsing and fragmentation of the pores. At the 10 % of the thickness of reduction the

porosity present in the Al-Si alloys very low. Which can be seen by the fig 5.4. At the 15 percent of reduction in thickness more grain elongation take place in the direction of rolling. The primary silicon congregates in microzone under the stress, and aluminium matrix flows into the porosities and connects in harness.

6.2 HARDNESS

The hardness increases with increasing the percentage of Si in Al-Si alloys and also the hardness of spray cast is higher as compared to that of I/M cast. The increase in hardness of Al-Si alloys with increase in Si content occurs because Si is a harder phase as compared to Al-phase. Fine grain formation in spray forming (due to rapid solidification) leads to high hardness as compared to that of I/M cast alloys. The hardness increases with the increase in radial distance from the centre to the periphery. This increase in hardness can be explained as follows. Heat transfer occurs from the perform due to the convective as well as the conductive heat transfer. The convective heat transfer varies from centre to periphery due to the variation in gas speed with radial distance. The conductive heat transfer rate increases from centre to periphery with the decrease in thickness of the perform. This increase in heat transfer rate from centre to periphery leads to more decrease in grain size at the periphery. A higher decrease in grain size gives rise to higher hardness at periphery.

Figure 5.3 shows variation in hardness along the surface of performs. Since cooling rate at the surface was higher than that in the core, so hardness values here is slightly more. Hardness along the surface was increase we move away from the center. This can be explained by the cooling rate. As cooling rate at centre is slower as compared to that at edge of disc formed so grain formed at centre are coarser and those at edges are finer.

The effect of rolling on Vickers hardness of the spray deposited Al-Si. In the initial stages of rolling (i.e. up to 5%), there is not much significant increase in VHN. However, it increases significantly beyond 5% rolling. This is shown in Figure 5.9 Hardness was increase as the percentage reduction of rolling this is because of the collapsing of the pores. when the reduction in thickness is continue metal flow take place in the pores and hardness is increases.

6.3 DENSITY AND POROSITY

Fig.5.14 shows the effect of percentage thickness deformation by rolling on the porosity of the deposited Al-Si spray formed alloy. It can be seen that porosity in the Al-Si layer decreases less rapidly up to 5% thickness deformation (about 12% decrease from the initial value). Porosity in the Al-Si deposit decreases rapidly (about 66% decrease from the porosity value obtained after 10% deformation). The decrease in porosity is due to metal flow in the thickness and as well length direction. As a result the pores are removed by process involving pore elongation in the direction of rolling followed by fragmentation into several smaller size pores. This process of elongation and fragmentation of pores continues with rolling. When the width of the pores becomes very small, the opposite faces of the pores collapse, and the porosity is eliminated. Elongation, fragmentation and collapsing of pores occur simultaneously during rolling. Decrease in the porosity which results in increase of density due to the collapsing of the pores. As shown in fig 5.13.

6.4 WEAR PROPERTIES

The wear rate of spray formed materials varied as a function of applied load. The wear rate continuously increased with applied load in Al-Si alloy however alloy shows low wear rate at a lower applied load which increases rapidly at a load exceeding 25N. Coefficient of friction was almost constant which is shown in the graph with respect to the time.

Wear rate decreases as increase percentage of the Si. Because Si is the hardest phase as compared to the aluminium.

6.4.1 EFFECT OF LOAD

From figure 5.12 may be seen that wear rate is dependent on applied load and it increases linearly with increase in load for all alloys sample investigated. It may be also observed that wear rate increases with increases in applied load in three distinct regions i.e. mild wear, intermediate wear and severe wear in Al-Si alloy. The mild wear of the alloy occurs as the sliding generates frictional heat and causes oxidation of fresh mating surface in the working atmosphere. Once the oxide layer formed, spalling and fracture of the former takes place over the wear pin surface because the load levels are not so high as to cause deep penetration and deformation in the surface layers below the oxide

layer. as the sliding progresses, the oxide particles are removed from the test specimen and may partly fill the valleys formed in the mating surfaces and the rest will form wear debris.

6.4.2 EFFECT OF ROLLING

In Fig shows that wear rate is decreases with the reduction in the thickness of the samples this is because the after the rolling porosity is reduces in radial and thickness direction . Contact surface is increases as the reduction of thickness and hardness is increases so wear rate is decreases .

The following conclusion are derived from the work of the present investigation .

- Spray forming provides a considerable microstructural modification in Al-Si alloy. the primary α - phase exhibits an equiaxed grain morphology with dispersion of Si particle in matrix phase. average grain size of 35 μm , and near-uniform distributed silicon particles surrounding the Al matrix,
- Hardness increases as the reduction in thickness. The primary silicon congregates in microzone under the stress, and aluminium matrix flows into the porosities and connects in hardness.
- The increase in hardness of Al-Si alloys with increase in Si content occurs because Si is a harder phase as compared to Al-phase. Fine grain formation in spray forming (due to rapid solidification) leads to high hardness as compared to that of *I/M* cast alloys.
- Density of Al-Si alloys mainly depend on the Si amount present in the alloys because Si have higher density as compare to Al.
- In the initial stages of rolling (i.e. about 5% thickness deformation), the metal flow in the Al-Si deposit is mainly in the thickness direction resulting in the removal of porosity by rearrangement and restacking of spray deposited particles. Beyond this much deformation, plastic deformation becomes the predominant mechanism of densification during rolling.
- Wear rate increases linearly with increase in applied load in three distinct region; mild wear at low loads, intermediate wear at moderately high load and severe wear at high load.
- Wear rate decreases linearly as the percentage of rolling increases because hardness increases.

CHAPTER-8

RECOMMENDATIONS FOR FUTURE WORK

- The process parameters of temperature , pressure, deposition distance from nozzle to substrate, substrata material can be changed in order to attain the optimum properties for the spray form.
- Substrate can be rotary or tilting movement for obtaining different shape of preform.
- Al -Si alloys can be used as thermal management material with increase the amount of Si .
- The detailed study on the wear behavior of leaded Al-Si alloys can be done for different sliding speed and load up to 50 N.

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