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SPRAY FORMING OF AL-10Si-10Pb: A STUDY OF THEIR MICROSTRUCTURE AND WEAR CHARACTERISTICS

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ABSTRACT

The liquid immiscible alloys based on Al-Pb system are potential materials for application in automobile and aerospace industries. However, their processing by the conventional casting techniques is difficult due to liquid immiscibility in a wide range of temperature and com- position and also large density difference of the constituent phase.

In the past, several techniques different from conventional casting have been employed to prevent segregation of lead during freezing of the melt. Techniques based on ultrasonic vibration of melt, powder metallurgy, stir casting, space metallurgy, rheocasting, strip casting, melt spinning and spray forming, results in a uniform distribution of lead particles in Al matrix. However, some of these techniques are often associated with either a higher energy consumption or generation of coarse grain microstructures.

Among these techniques, spray forming possesses several advantages in effective micro structural control together with producing a near net shape preform in a less number or processing steps. In this process, the melt is superheated to a temperature above the liquid immiscibility region of the melt prior to atomization. Rapid cooling associated with solidification of atomized droplets and a turbulent fluid flow condition on the deposition surface minimizes the separation of the Al and Pb-rich phases. However, a high melt temperature result in rapid coarsening of Pb particles in this process. The present investigation reports a modification of the spray forming process to avoid coarsening of Pb particles in Al-10Si-10Pb alloys. The micro structural features of the spray formed alloys and their resultant wear characteristics are reported.

The wear testing of spray formed alloys were investigated using a pin on disk type wear testing machine. The standard wear test procedure was followed for evaluating the wear rate for different load ranging from 10 to 90 N at a constant sliding speed of 1.0 ms^{-1}. All the tests were carried out in dry sliding conditions and at room temperature. The worn out surface and debris particles were preserved periodically for further examination. The wear rate was observed in the range of 1 to $6.5X10^{-12}m^3m^{-1}$ and the coefficient of friction was found to be 0.4.

Keywords - Spray forming, Al-10Si-10Pb, Microstructure. Wear characteristics.

1 INTRODUCTION

The principles of the spray deposition process were developed by Singer at the University of Swansea in the early 1970s. Spray forming is a metal forming process that spews molten, atomized metal at a rotating substrate to form a metal ingot or billet. The high solidification rate of the process results in a well-defined microstructure. The process produces material similar to that achieved by powder metallurgy at a fraction of the cost. Billets or bars of different shapes are possible, depending on the substrate shape. The process is applicable to a wide variety of alloys.

Al-Pb alloys are gaining more importance in engineering applications like aerospace, automobile, etc., due to its wide range of physical, mechanical and tribological properties. However conventional cast materials exhibits poor properties compared with spray formed materials. Previous literatures have shown that the particle size, microstructure and mechanical properties are good when Al-Pb based alloys are manufactured by spray deposition process. Gupta and Lavernia studied the microstructure of hypereutectic Al – 17Si – 4.5%Cu alloy and their analysis revealed that spray atomization and spray deposition processing leads to significant refinement of microstructure as compared to its as cast counterpart. They observed the microstructure characteristics as follows: (a) an equiaxed grain size (b) Unconverted porosity (c) nearly spherodized secondary phases.

2 SPRAY FORMING PROCESS

The basic parts of a spray forming unit are a melting unit or a furnace, a gas atomizing unit and a preform unit as shown in Fig.1. A metal melting unit is basically a furnace for melting alloys ranging from aluminium to steel and cast iron. It is fitted with a nozzle through which metal will be sprayed.

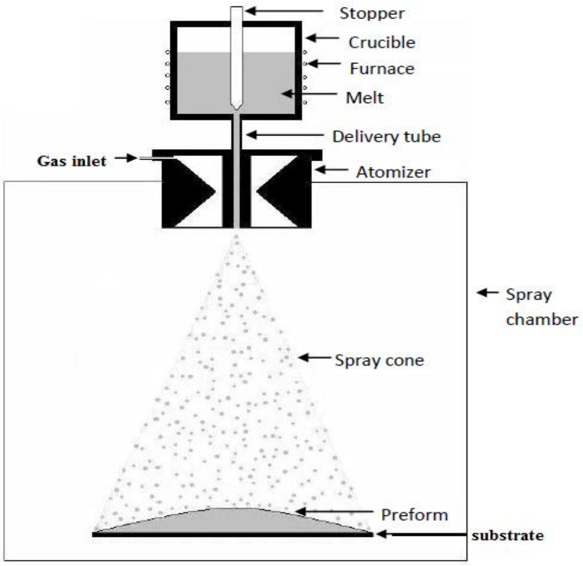

Fig 1: Spray forming process

The particulates can be either incorporated in atomization zone Fig.2 (a) During melting the alloy Fig 2(b).

Spray forming is unique solidification process in which metal melt is atomised by inert gas in to droplets of 10-200 microns in size, flying at subsonic speed into deposit substrate. During the flight the droplets are rapidly cooling with a cooling rate between 100-100,000 degrees per second in a controlled way so that, solidification of the metal is not dependent on the temperature and or the thermal properties of the deposition surface like a mould. The particles arriving at the mould are in such a condition that welding to the already deposited metal is completed and no interparticle boundaries are developed.

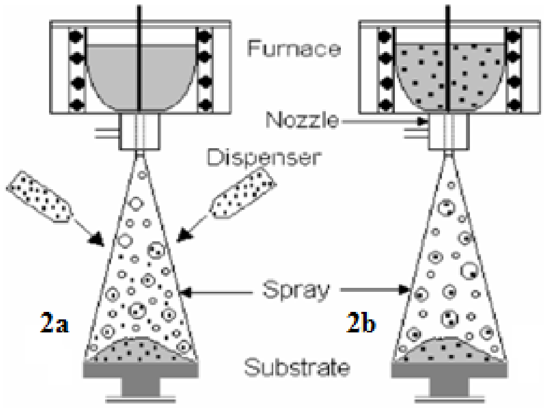

Fig 2 (a): Particulates incorporated during atomization, (b): Particulates incorporated during melting the alloy.

As result high quality metals are made with fine equiaxed and homogeneous microstructure. These features are special prominent in making high alloy metal components like for examples die inserts, tooling heads.

3 SPRAY DEPOSITION OF Al-10Si-10Pb

The details of spray forming set-up are described elsewhere. In brief, the process employs an annular convergent–divergent nozzle, with a throat area of 20×5 mm2 and an exit to throat area ratio of 3: 1, for atomization of the melt. In this process, the gas interacts with the melt stream at the tip of a flow tube concentric to the gas flow channel to promote atomization of the melt. The resultant spray of droplets is deposited on a steel substrate in an environmental chamber connected to a cyclone to collect the over spray powders. A schematic illustration of the deposition process is shown in **Fig 3(a).**

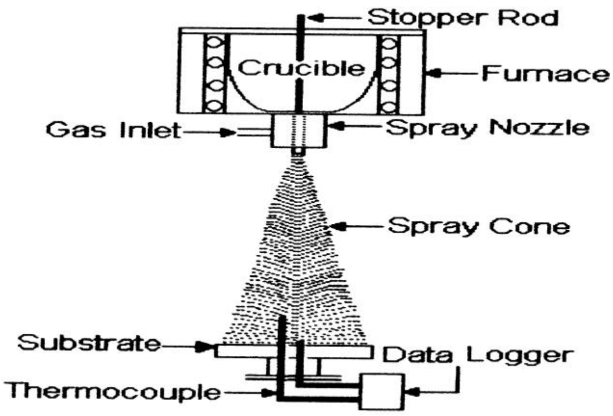

Fig 3(a): Spray deposition process.

Fig 3(b): Epiremental set up used for atomization and deposition processing.

The composition of alloy consisted of Pb–12% Sn–12% Sb–1.5% Cu with traces of arsenic (composition is given in wt%). In each experiment 2×5 kg of the alloy was charged in a graphite bonded fireclay crucible. The melting was carried out in a resistance heating furnace under nitrogen gas atmosphere. The temperature of the melt was measured using a chromel–alumel thermocouple connected to a temperature recorder. A melt superheat of 200°C was ensured to stop the premature freezing of the melt. The details of the process variables are listed in Table 1.

The melt was atomized by nitrogen gas at an atomization gas pressure of 12 MPa and nozzle to substrate distance of 0.45 meter. The thermal profile measurement was carried out during this experimental condition only. In another set of experiments the gas pressure was reduced to 1 MPa and nozzle to substrate distance of 0.25 meter. The melt flow rate was obtained by the mass of the charge in the crucible and duration of atomization. The gas flow rate was measured using a gas flow meter.

4 MEASUREMENT OF THERMAL PROFILE

Two chromel–alumel thermocouples were inserted through a hole in the substrate. These were centered along the axis of the spray. The hot junctions of thermocouples were positioned at a distance of 2 mm and 10 mm above the surface of the substrate. Thermocouple wires were insulated in alumina sheaths to avoid their contact with the metal substrate. The spray nozzle and substrate were leveled to align the thermocouples with the axis of the melt spray. The outputs of the thermocouples were recorded during and after spray deposition using a Data Acquisition System with a response time of one sec.

5 PROCESS VARIABLES

In the process of spray forming, there are number of independent and dependent variable which major influence the metallurgical and other integrity of a spray deposit. Some independent process parameters are as follows: Gas Pressure (in range of 0.05-1.0 MPa), Molten Metal Superheat (typical range 10-200^{o}c), Melt Flow Rate (90-120gs^{-1}) Substrate Position, i.e. the distance between the gas nozzles and the substrate (300-350mm or more) Substrate motion, which includes substrate rotation speed, withdrawal rate and tilt angle.

These independent parameters can be directly controlled during the process and affect a number of dependent process parameters. There are number of pre-set parameters which cannot be changed during the operation i.e. Type of atomizing gas (Nitrogen and Argon) Diameter of melt delivery tube the atomizer design Substrate material

On the other hand, if most of the droplets in the spray and/or on the surface of the deposit are liquid, the metallurgical properties of the spray casting will be similar to those of a conventional casting. Thus, the ideal amount of liquid must be between these extremes. Therefore, the process parameters should be controlled to produce the optimum mixture of liquid and solid droplets.

6 EXPERIMENTAL PROCEDURE

To investigate the micro structural features and the Pb particles distribution in the matrix alloys, one composition of Al-10Si-10Pb base alloy was used. Several spray forming runs have been conducted with varying nozzle to substrate distance, different melt temperature and atomizing gas pressures. The composition and other details of the processing parameters are given in the Table I and II. Commercial grade lead used in these experiments for Pb addition. The atomizer used was an annular confined type convergent divergent nozzle. A graphite crucible was used for bottom pouring system. A required quantity of base alloy was charged in the crucible with tapered stainless steel stopper rod and melted in a resistance heating furnace. During melting, temperature of the alloy in the crucible was continuously monitored by a chromel – alumel thermocouple.

A gas regulator controlled the flow rate of the inert gas for atomization. Lead granules were introduced in to the vortex of the melt in the crucible from the top of the furnace just prior to atomization and continued. Following atomization the droplets were allowed to deposit on to the substrate positioned at a fixed distance below the atomizer. The bell shaped preform with a height of 30 mm and a diameter of 200 mm at the base was obtained. Samples cut out from the different regions of the preforms were prepared by standard metallographic techniques. These were examined by optical microscopy and scanning electron microscopy.

Element % base alloys	Wt% alloy
Si	10
Cu	0.5
Fe	0.15
Mg	0.2
Mn	0.01
Al	Remaining
Pb	10

Table 1: Alloy composition

7 PROCESS DESCRIPTION

The spray forming process employed in the present work consisted of a spray nozzle assembly, an atomization chamber and control system to maneuver the gas flow rate and movement of the deposition substrate. The spray nozzle included a gas flow channel of convergent- divergent configuration centered around a melt delivery tube. The schematic diagram of the deposition process is shown in Fig. 6. As the gas is passed through the nozzle considerable suction is created at the end of the melt delivery tube.

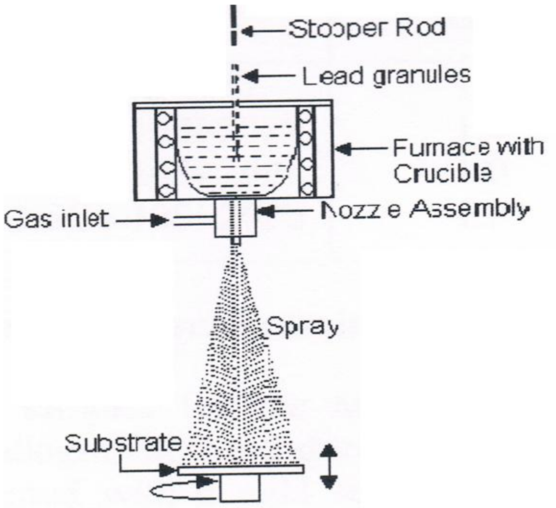

Fig 7: Schematic diagram of spray forming process.

The lead either in the form of granules or rod is introduced in controlled volume in to the molten Al-alloy the center line of the crucible during atomization. The vortex created in the melt due to suction pressure is made use of to direct the stream of high density lead concentric to the stream of Al-melt towards the inlet of the atomizer.

The gas melt interaction in this configuration takes place at the tip of the flow tube to promote atomization of the melt. This results in a high efficiency of the melt atomization and subsequent generation of small size droplets in the spray. The Pb and Al –rich droplets are formed in the spray which is subsequently deposited over a copper substrate centered along the spray axis at a predetermined distance. The flowability of the melt is enhanced in this process due to addition of high density lead. The composition of lead in the preform can be independently controlled by varying the amount of lead in the melt of Al –alloy. The overspray powders are collected after the experiment.

The base alloy composition used in the present work consisted of Al -0.5% Cu-10% Si, 0.15% Fe, 0.20% Mg,0.01%Mn (composition in wt%). The alloy was prepared in an induction melting furnace under the cover of an inert gas atmosphere. The melt was subsequently cast in to form of ingots. These were brought and remelted during the spray deposition experiments. Lead in the form of granular was added to the melt in some of the experiments to control the size and size distribution of Pb particles in the matrix phase of the base alloys.

The melting was carried out in a graphite crucible using a resistance heating furnace. In each run, 2.0Kg of the alloy was charged in the crucible provided with an opening at the bottom. The opening in the crucible was sealed initially during melting of the alloy with a stainless steel stopper rod. The melt flow was promoted through the crucible by lifting the stopper rod.

During melting, the temperature of the melt in the crucible was continuously monitored using a chromel-alumel thermocouple connected to a temperature controller. A gas regulator controlled the flow rate of the N2 gas for atomization. Lead granules of 3 to 5mm diameter were introduced in to the melt along the centerline of the crucible from the top of the furnace during atomization of the melt. The atomization was carried out at a N2 gas pressure of 1.0MPa. The details of the process variables are given in Table 2. The spray was subsequently directed towards the copper substrate centered along the spray axis to achieve a disc shape preform of 200 mm diameter with a height of 30mm at the center. The nozzle to substrate distance was 0.45m in this experiment. The over spray powder particles were collected for the microstructure analysis. Several samples were machined from different locations of the deposit for microstructure examination and wear testing.

Alloy Composition	Al -10Si-10Pb
Gas pressure MPa	1.0
Gas/melt flow ratio	0.89
Melt Temperature,^{0}c	750
Deposition Distance ,m	0.45

Table 2: Process variables employed during spray forming.

8 MICRO STRUCTURAL EXAMINATION AND WEAR TESTING

The samples for the microstructural investigation were prepared following the standard metallographic procedure of grinding and polishing. Over spray powder particles were mounted with a cold resin to facilitate metallographic preparation. All the samples were etched in a Keller's reagent. The micro structural examination setting acrylics was carried out in a metallux-3 optical metallograph. Several samples including that of the wear test specimens and debris particles were also examined in a Joel 840, a scanning electron microscope operating at 15kV.

The wear testing of spray formed alloys were investigated using a pin on disk type wear testing machine. It consists of a hardened steel disk with hardness value of 52Rc and a specimen holder. The specimen consisted of a cylindrical pin with 8mm diameter and 20 mm length. The load on the specimen was applied by placing a load on the opposite side of a fulcrum of the lever to attach to the specimen holder.

The reduction in the length of the wear sample was measured through a linear variable differential transducer (LVDT) attached to the other side of the fulcrum at the same distance at which specimen was placed from the fulcrum in opposite side. The frictional force on the specimen was measured using a load cell. This measured the force component of the moment acting on the specimen but at the opposite end of the fulcrum.

The standard wear test procedure was followed for evaluating the wear rate for different load ranging from 10 to 90 N at a constant sliding speed of 1.0 ms-1. The disc surface was cleaned with acetone before each experimental run. All the tests were carried out in dry sliding conditions and at room temperature. The worn out surface and debris particles were preserved periodically for further examination.

9 RESULTS AND DISCUSSION

9.1 Micro structural Features

The microstructure of Al-10Si-10Pb alloy invariably exhibited equiaxed grain morphology of the primary α-phase both at the central and peripheral regions of the spray deposits Fig.9a. Considerable modification was observed in the morphology of the Si particles as a constituent of the eutectic phase as shown in Fig.9b. The globular shape of the Si particles was observed either along the grain boundary or in the intra- granular regions.

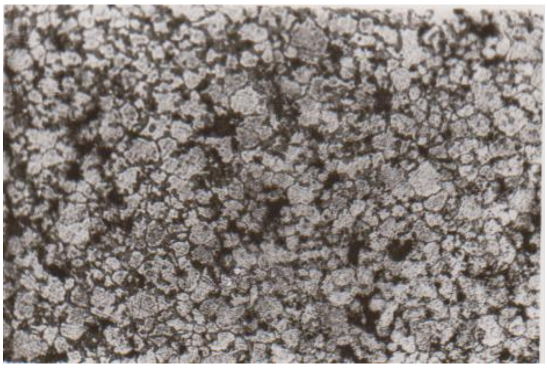

Fig:9(a)

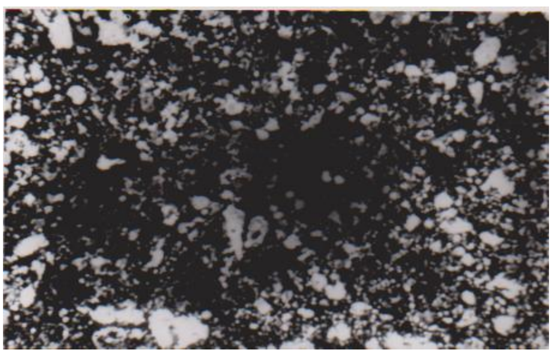

Fig:9 (b)

Fig 9: Microstructure of spray deposited Al-10Si-10Pb alloy showing (a) equiaxed grain morphology, (b) particulate morphology of Si phase.

The size of the Si particles was often observed to be less than 2 micron. Several submicron size particles were noticeable in the intra- granular regions. However, considerable refinement in the size of Si particles was observed to be characteristics feature of the deposit in the peripheral regions. The micro structural feature of Al-10Si-10Pb alloy also exhibited similar morphology of the Si phase. The effect of deposition distance on the micro structural features of this alloy composition was investigated. The results indicated that, at a deposition distance of 0.45m, the lead particles in the central region of the deposit exhibited ultrafine particles of lead coexisting with some of the larger particles Fig. 9c. The top region of the deposit for this deposition distance also indicated needle shape morphology of the Si phase arising as a result of slow cooling rate of large liquid pool generated on the top surface of the deposit Fig.9d.

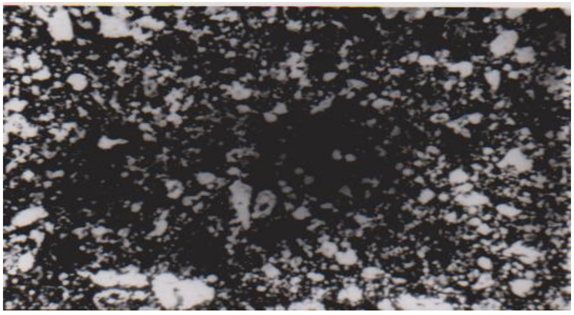

Fig: 9(c)

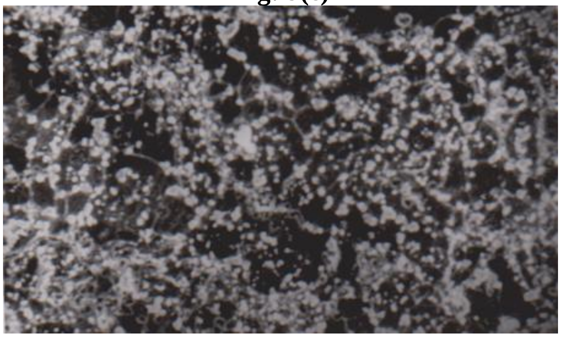

Fig: 9 (d)

Fig.9. Microstructure of Al-10Si-10Pb alloy spray deposited at a nozzle substrate distance of 0.45m showing (c) debris particles of Pb, (d) acicular morphology of eutectic Si.

The alloy that was spray deposited at a deposition distance of 0.45m exhibited several interesting microstructure feature. A bimodal size distribution of lead particles was observed in the bottom region of spray deposit. The larger particles corresponded to 10 to 25 micron size coexisting with smaller lead particles of 0.5 to 1 μm size.

The microstructure also indicated considerable variation in size of lead particles in the bottom and top region of the deposit as shown in Fig. 9 e, f. some of the central region of the spray deposit also indicated elongation lead particles along the grain boundaries of the primary phase with coarse irregular shape particles decorating the grain junctions Fig.9g. Since the freezing temperature of Si phase is higher than that of Pb, some of the region of the deposit revealed Si particles surrounding by lead Fig.9 (h). Similar microstructural feature were observed in the spray deposit with variation in deposition distance during spray forming.

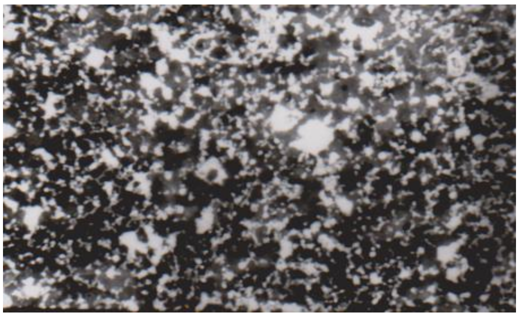

Fig:9(e)

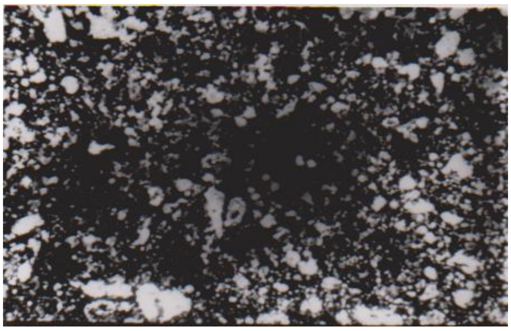

Fig:9(f)

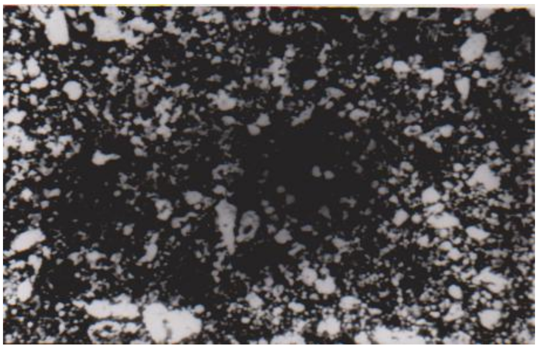

Fig:9(g)

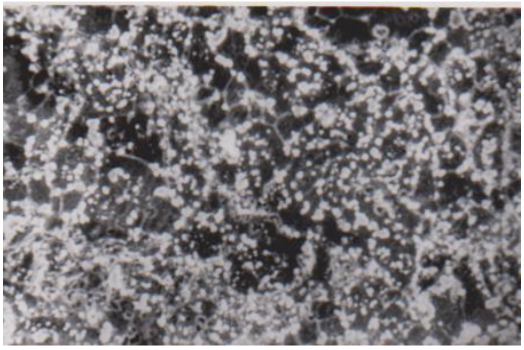

Fig:9(h)

Fig 9. Al-10Si-10Pb alloy at deposition distance of 0.45m showing (e) bimodal size distribution of Pb, (f) ultrafine Pb particles in the bottom region of the deposit, (g) stringers of Pb particles in the central region and (h) Si particles surrounded by Pb phase.

An increase in the deposition distance to 0.55m results in a uniform dispersion of ultrafine lead particles in the matrix as shown in Fig9 (i). However, the grain boundaries of the primary phase could not be clearly delineated in this region. In addition, large amount of porosity was observed in the deposit Fig.9(j).

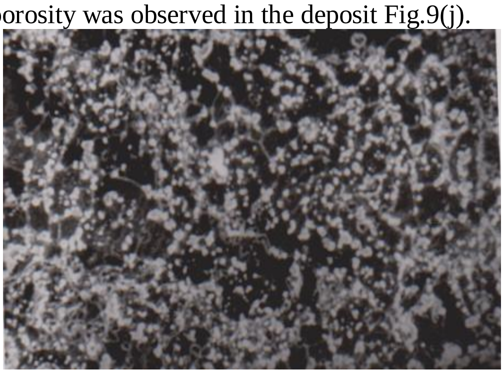

Fig:9(i)

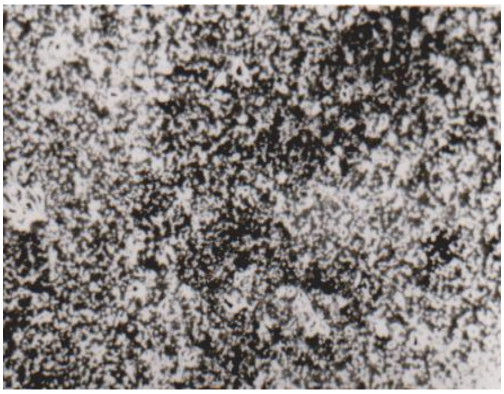

Fig:9(j)

Fig.9. Micro structural characteristics of Al-10Si-10Pb alloy produced at a deposition distance of 0.55m showing (i) uniform distribution of ultrafine Pb particles, (j) absence of distinct grains.

10 MICROSTRUCTURE OF POWDER PARTICLES

The overspray powder particles exhibited cellular and dendrite morphology of the primary phase in Al-10Si-10Pb alloy. The lead particles were observed to be present in the inter-cellular and inter-dendrite regions. In smaller particles, the volume fraction of the cellular region was observed to be more than that of the larger particles Fig 10 (a), (b). The secondary dendrite arm spacing was observed to be dependent on the size of the particles.

The solidification structure often indicated nucleation event occurring on surface and center of the particles. The cooling rate of the powder particles estimated from the secondary dendrite arm spacing was observed to vary from 10^3 to 10^4 Ks^{-1}, the higher cooling rate corresponding to the small size particles. In addition, several powder particles revealed distribution of spherical shape Al particles in the Pb –rich matrix phase as shown in Fig. 10 c. This result indicates presence of two different type of particles one rich Al and other in Pb.

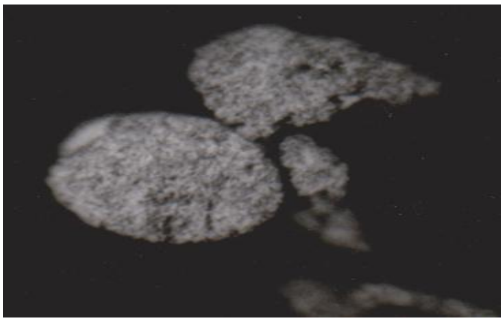

Fig:10(a)

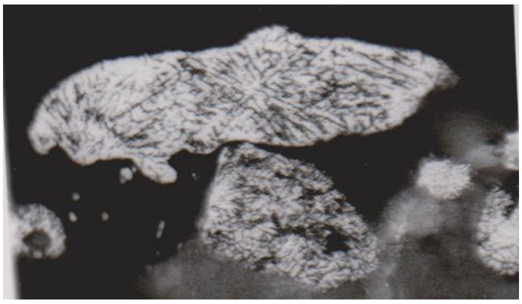

Fig:10(b)

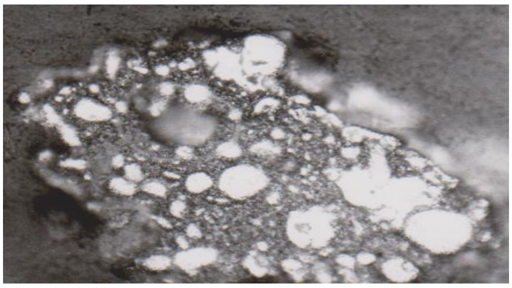

Fig:10 (c)

Fig 10: Microstructure of Al-10Si-10Pb alloy showing (a,b) cellular-dendritic morphology of the primary α -phase in two different size powder particles (c) fine Al-particles(bright) in the Pb-rich matrix phase(dark).

11 WEAR CHARACTERISTICS

The wear rate of spray formed alloy was observed to increase with an increase in applied load. However the rate of Al -10Si-10Pb alloy was often higher than that of alloy containing 20% lead. A transition in wear rate was observed at an applied load of 50N. This feature was consistently present in alloys either containing leads or without lead Fig 11(a). The coefficient of friction was found to sharply decrease up to an applied load of 30N. Subsequently, this value remained constant in a wide range of applied load Fig 11(b). A wide difference was noticeable in the value of coefficient of friction of these alloys. An addition of lead resulted in a value of the coefficient of friction of 0.2 against a value of 0.35 for the alloy that did not contain lead.

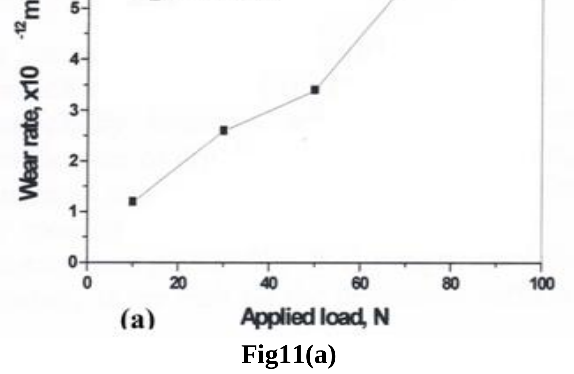

Fig11(a)

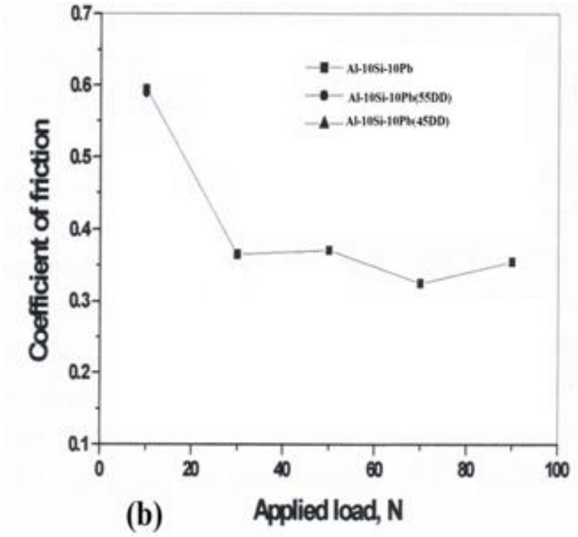

Fig11(b)

Fig11(c)

Fig11 (d)

Fig11. Variation in (a) wear rate, (b) coefficient of friction of spray formed alloys as a function of applied load, and Morphology of worn out surfaces of wear test specimen showing (c) smearing of Pb particles (d) wear debris.

Scanning electron microscopy of the worn out surface of the test pins indicates smearing of lead particles in alloy containing lead even at a low applied load Fig.11(c). in contrast to irregular shape of oxide particles and scoring on the surface of the test pin of the base alloy.

12 DISCUSSION

The microstructural features generated during spray deposition process depends on a combined consequence of solidification path of the alloy system during atomization as well as during spray deposition process. It is well known that an Al-Si system shows an asymmetric coupled zone below the eutectic temperature. A high cooling rate achieved during solidification of atomized droplets results in considerable undercooling of the melt. This behavior increases the volume fraction of the primary α-phase with its cellular and dendritic solidification morphology in powder particles.

As the droplets in semi-solid or semi –liquid state impact on the deposition surface the dendrites are fragmented in to debris particles. This feature is also promoted as a result of turbulent fluid flow conditions on the deposition surface. Subsequent solidification of the remaining melt takes place from various nucleation sites provided either by the debris particles or fully solidified fine particles. As a consequence of enhanced nucleation and constrained growth of the phases on the deposition surface, the solidification interfaces converge at common boundaries.

This mode of solidification of the spray-deposit results in equiaxed grain morphology of the primary α-phase. The growth of silicon as a constituent of the eutectic phase is restricted in highly non equilibrium solidification condition that prevails on the deposition surface. An observation of the particulate morphology of Si particles, as a constituent of the eutectic, indicates deviation from the equilibrium solidification condition.

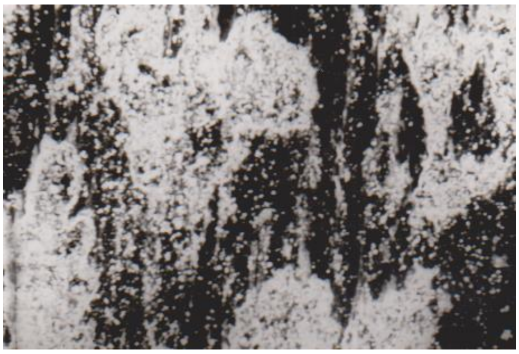

Fig12 (a)

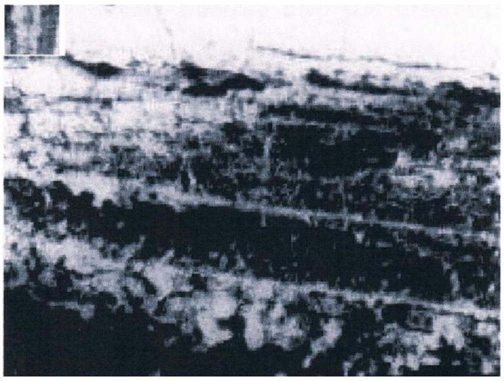

Fig12 (b)

Fig12: Morphology of worn out surfaces of wear test specimen showing (a) smearing of Pb particles, (b) irregular oxide particle.

An altogether a different solidification path is followed during spray forming of liquid immiscible alloys. Atomization of the melt from the temperature above the liquid immiscible boundary initially results in separation of Al and Pb rich liquid during solidification of droplets. However, high cooling rate associated with solidification of droplets minimizes coarsening of Pb- rich droplets.

Once the temperature of the melt approaches to the monotectic temperature of the alloy on the deposition surface, the primary Al phase nucleates and grows. The Pb rich liquid pool further breakdown in to small size droplets due to agitation of the melt on the deposition surface. In this case, the morphology of Pb particles depends upon the solidification rate of Pb rich droplets. A slow cooling rate results in spheroidization of the droplets. Alternatively, their irregular shape is preserved during high cooling rate. As a consequence of this, the morphology of lead particles varied in different region of the deposits that undergoes a variation in cooling rate.

However, in the present investigation a modified spray forming technique was used to incorporate Pb at a relatively low temperature in the vortex created in the molten Al-alloy during atomization of the melt. The molten Pb and Al are dragged by the suction pressure developed at the tip of the flow tube and delivered to the gas stream to promote atomization of the melt. Consequently, droplets of Al and Pb-rich liquid form and interact with each other in the spray as these are directed towards a deposition surface.

Solidification of Al- rich droplets develops a cellular- dendritic morphology of the primary phase whereas Pb-rich droplets give rise to fine presolidified particles of Al dispersed in the Pb rich matrix. The evidence of this feature is clearly present and occurs between the Al- and Pb rich droplets on the deposition surface. The high volume fraction of solid on the deposition surface in contribution from the Al-Si droplets and the large liquid fraction arise from the Pb-rich phase. The liquid Pb spread in to thin sheet once they impact on the deposition surface.

The melt agitation arising from the momentum of the depositing droplets create a turbulent fluid flow conditions and shearing action in the semi molten zone on the deposition surface. This effect facilities further disintegration of the liquid Pb in to fine droplets with their different aspects ratio. The small size droplets of Pb are subsequently pushed in to the inertial voids generated due to solidification of Al-rich phase. The Pb – rich droplets with a low aspects ratio rapidly spherodized giving rise to spherical particles of Pb on solidification of the melt. On the other hand, the liquid film with a high aspects ratio retains the elongation shape of Pb particles. This feature has been observed in the microstructure of the spray deposit.

13 CONCLUSIONS

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

Spray forming provides a considerable microstructural modification in Al-10Si-10Pb alloy. The primary α-phase exhibits equiaxed grain morphology with dispersion of Si and Pb particles in the matrix phase. Additionally Si as a constituent of eutectic phase shows a particulate morphology in contrast to its characteristic needle shape morphology in the as cast alloys.

The grain size of the primary α -phase varies from 10-25µm depending upon the process variables employed during spray deposition. Similar variation is observed in the size of Si and Pb particles. The size of the Si varies from 0.5 to 5µm and that of the Pb particles from 0.1 to 25µm. The micro structural features of the spray formed alloys influence their wear properties. The wear rate and coefficient of friction of the alloy containing 10% lead is observed to be lower.

Examination of the wear test specimens reveal a continuous film of lead smeared over the surface. On the other hand irregular shape debris particles are observed in the alloy free from lead.

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