PREPARATION OF SOME ALUMINIUM-BASE ALLOY POWDERS BY ATOMIZATION AND STUDY OF THEIR SINTERABILITY

A DISSERTATION
submitted in partial fulfilment of the
requirements for the award of the degree
of
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in
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(PHYSICAL METALLURGY)

By
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CERTIFICATE

Certified that the dissertation entitled "PREPARATION OF SOME ALUMINIUM BASE ALLOY POWDERS BY ATOMIZATION AND STUDY OF THEIR SINTERABILITY" which is being submitted by Shri Suresh Chand in partial fulfilment for the award of the Degree of Master of Engineering in Metallurgical Engineering (Physical Metallurgy) of the University of Roorkee, Roorkee, is a record of his own work, carried out by him under my supervision and guidance from 3rd January 1972 to 15th September, 1972.

The matter embodied in this dissertation has not been submitted for the award of any other degree.

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(SURESH CHAND)

ABSTRACT

In this project, the installation of close type atomizer for continuous production of powder is made. Aluminium-tin and Aluminium-copper prealloyed powders of different compositions have been prepared and the characteristics (size distribution and sinterability via. hardness and densification) of powder are obtained. Addition of tin or copper helps in the production of finer powders than is possible with aluminium alone.

Under sintering tests, effect of grinding for removal of alumina film and thereby for improving sinterability is tested. Sinterability of prealloyed powder and unalloyed powder mixture is compared. Results show that prealloyed powders have better sinterability. Apart from this compact of aluminium-tin-copper powder mixture are sintered. Results show addition of copper to Aluminium-tin powder mixture improves the sinterability.

CONTENTS

CHAPTER - I

INTRODUCTION

For producing complicated shapes of typical materials with typical properties, powder metallurgy has its important role. Basically it consists of production of metal powder, compaction to desired shape with the help of die and punch and sintering to get Strength. Out of all those steps the production of metal powder is the one least developed.

Thermodynamically, Difference in Solid metal block and the metal powder heap lies in their specific surface energies, where the latter one has more specific surface energy as compared to the metal block. Thus powder making from solid metal block will involve energy for generation of new surfaces. According to the modes of transfer of energy, there are various methods of producing metal powders such as electrolytic deposition, precipitation, reduction, crushing or grinding, and, atomization, where electricity chemical potential, mechanical pressure and fluid dynamics are used as modes of transfer of energy respectively.

The most significant development in production of metal powder, in recent years, has been that of atomization. Basically, in principle it involves forcing the molten metal through a small orifice and breaking up the stream by a jet of stream of compressed air, inert gases water or sometimes liquid petroleum products have been employed.

The controlling factors in the atomization are :

1. Nozzle Size and design
2. Temperature of metal
3. Rate of flow of molten metal
4. Temperature of the atomizing medium
5. Inlet pressure of the atomizing medium

The particles produced tend to have globular shape Since the liquid metal freezes almost instantaneously. Mostly the size range of the powder produced is 20 - 400 microns. The oxygen content of the powder is very low being usually in the range of 0.2 - 0.3%. This is due to rapid cooling and formation of very thin protective oxide surface film.

Where inert gases have been used in the process, the metal has been found to be highly reactive and the powder, therefore, should be suitably protected from oxidation by atmospheric oxygen.

The suitability of atomization for producing alloy powders has led to its extension to the higher melting point ferrous and nichel base alloys. Early attempts to use systems similar to that described by Thomson[1] were unsuccessful because of rapid failure of the refractory nozzle. Other methods for the atomization of ferrous and other high melting point alloys have been reviewed by Walhimson[2] who also describes a method which has been highly successful for alloy steels and nickel base alloys. One of these the D.P.G. method[11] (deutsche pulver metallurgische Gesells chaft process), in which a number of rapidly rotating blades break up a stream of molten metal. The resulting droplets are rapidly cooled by a jet of water which surround the metal stream. This method was used for number of years but was eventually abandoned, owing to the inherent disadvantages of metal freezing on the blades and excessive oxide film formation on the particle.

One of the greatest advantage of atomization is that it can be applied to any metal and alloy, that can be melted irrespective of mechanical properties of metal and alloy under consideration whereas, in any other process of powder production one has to consider the mechanical properties also.

The importance of this process is related not only to the great variety of metal and alloys to which it can be applied, including very high melting point metals, but also to the fact that it is comparatively economical, yet yielding good quality of powder. Powder produced by the method are not reactive and, although the atmosphere may be air, only superficial oxidation takes place whereas, powder produced by cementation and electrolysis and certain other methods have a serious problems of oxidation and harmful contaminations.

In the case of atomization process, apart from the oxygen content there are no chances of other types of impurities getting incorporating into the powder during the process of its production. Whereas in case of other methods many other objectionable impurities also appear during the process such as remains of electrolyte (in electrolytic deposition), erosion of containers (in mechanical process), impurities picked up at high temperature (in high temperature reduction process), from the surroundings etc. etc.

Rate of production by this process is very high as compared to other methods, For these reasons this method has been found very much suitable and economic for industrial production.

However, the main limitation of this process is the wide distribution of the size in the powder produced. If one wants to produce powder of a particular size simultaneously he will get other size also. Finest powder that can be obtained is of the order of 10^{-4} mm (350 mesh) while in other process we can get powder of very narrow size distribution and almost as fine as 400 mesh. However, by controlling the various design factors it is possible to enhance the percentage of particular size powder.

Due to the working difficulties at high temperature, normally refractory metals can not be atomized.

CHAPTER - 2

LITERATURE SURVEY

Atomizer

As the mechanical strength of liquid metal is lower than that of solid metal, less difficulty will be experienced in its disintegration, and that is why the disintegration of melt is a convenient and popular technique of powder production, Atomization. Liquid disintegration in this, is carried out with the help of atomizer. Atomizer mainly consists of melting furnace to get the solid metal into liquid form and fluid nozzle to disintegrate the liquid stream into drops. There are two main types of atomizer which are as follows :

1. Closed type atomizer
2. Open type atomizer

The closed type atomizer[3], which has been shown in fig. 1 is usable only for lower melting point metals at least on a small scale, because of danger of freezing of liquid metal in the delivery refractory tube, such atomizers have a special advantage that the stream of liquid metal is conducted accurately to the point at which the atomization starts. They are, therefore, easy to use, consistant in operation and can be operated in hori-

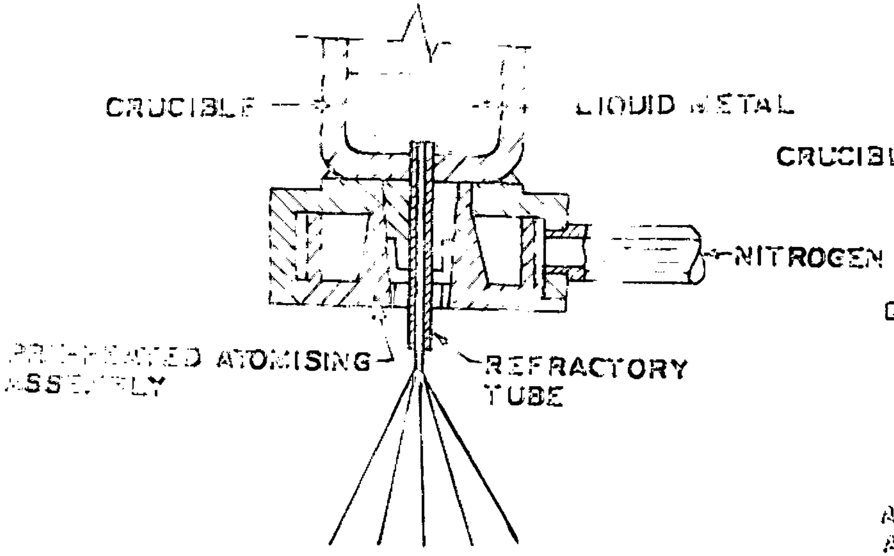


FIG. I CLOSED TYPE ATOMISER

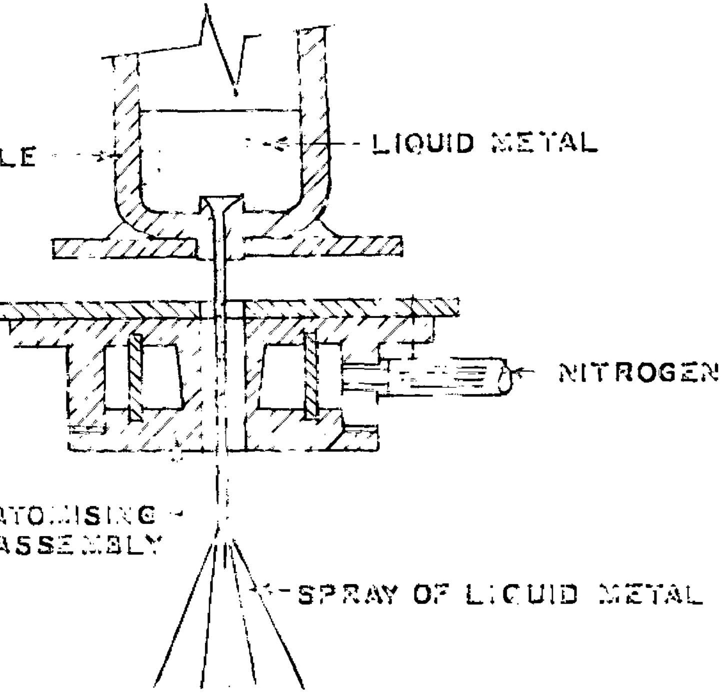


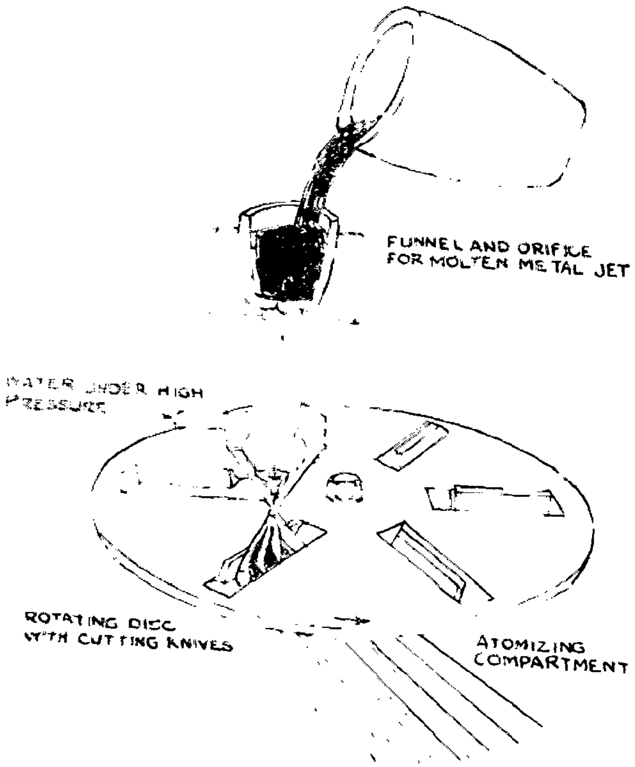
FIG. 2 OPEN TYPE ATOMISER

zontal and vertical positions. Unfortunately such nozzles when used, are to be preheated to the temperature of liquid metal to avoid choking of a delivery tube. This limits its use to only low melting point alloys.

Open type atomizers[3], shown in fig. (2) are more versatile. The salient feature of this is that a stream of liquid metal is allowed to fall by gravity into the atomizing zone so that the atomizer is not in contact with the metal stream and can be maintained as desired temperature of the gas. There are various difficulties in the operation of open type atomizers. The first difficulty is that the molten metal stream accelerates as it falls freely thus becoming smaller in diameter and finally divided into large drops. Atomizing must occur before this break up of the molten metal stream. The other difficulty is that the metal stream is easily deflected by turbulance in the surrounding gas envelope and it may waver before reaching the atomizing zone. Any major deflection of the metal stream causes oscillation and assymetry in the shape atomized particles finally produced.

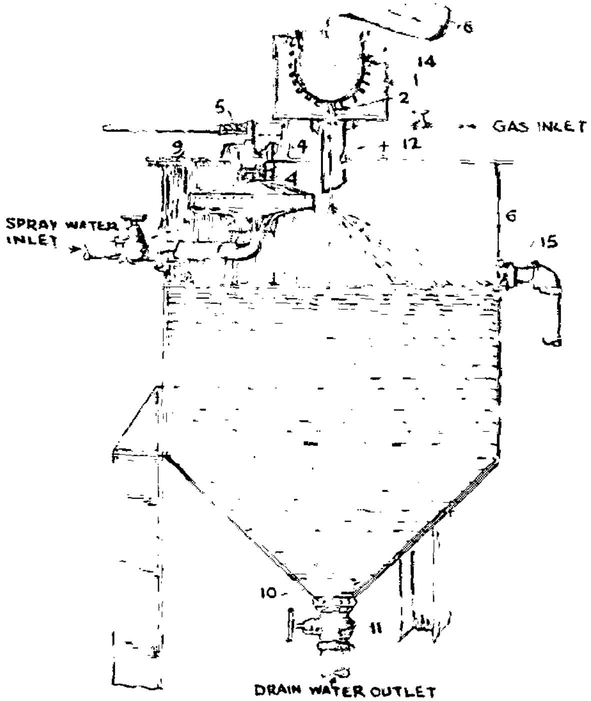
Effort has been made to reduce this deflection by minimising the distance between the metal stream outlet and atomizing zone. The work on this has been done by A.R.E Singer[3] at University of Swan Sea during his work on spray rolling of metal strips from powders produced by the process of atomization. He reduced this distance to approximately 25 mm. The shape of the outlet of liquid metal from the crucible and its finish effect the flow of the stream. The smooth outlet causes uniform stream and less turbulence.

Other type of atomizer, one of those is known as D.P.G. Machine has been shown schematically in fig. 3. In this molten metal flowed from a ceramic nozzle set in a tundish on to a set of rotating knives, the speed of which generally varied between 1500 and 3500 r.p.m. Chilling of the globules of metal and cooling of the knives and the apparatus was affected by means of an annular cone of water issuing around the metal stream. As the pressure of water varied between 60 to 120 psi, It is possible that some granulation of the metal was affected by the water stream itself. The particle size of the powder produced depended upon a considerable number of variables, including the temperature of the metal, the height of fall of the metal before it hit the knives, the pressure of the water, the speed of rotation and the shape and disposition of the knives.



Diagrammatic sketch of Liquid Disintegrator of Deguss Treatise

FIG. 3



Diametrical representation of Liquid Disintegrator of Comstock type.

FIG. 4

Though this machine was very much useful in meeting a war time emergency requirements in Germany and produced a large tonnage of metal powder, now a days it has become obsolete due to number of disadvantages involved in it. Not only were these operational and maintenance difficulties, which must arise in any apparatus rotating at high speed in contact with molten metal. The metal also tended to freeze on to the knives especially when copper was atomized and in particular the product got oxidized and tended to be very coarse.

In America in 1944 Gregery J. Comstock[13] adopted a similar but some what different line of approach. Instead of employing rotating knives with all the associated problems of metal sticking, an apparatus was designed more or less on the principle of a "rotating garden spray" in which rotating jets of water replaced the rotating knives. At first sight it might seem that the apparatus would perform very inefficiently because the energy of the water system is expended in hitting the metal stream only for a small part of the total are of rotation. However, as the primarily produced metal globules take an appreciable period before they freeze, it is probable that each globule receives several hits by several jets, and thus the overall efficiency is thereby somewhat increased. The apparatus has been shown in fig. 4. The machine also possess the same disadvantage as that of D.P.G. machine.

Nozzle Design

Different nozzles were suggested by various workers for production of metal powders. The design of nozzles depend upon number of factors like

(a) The type of metal being atomized

(b) The temperature of atomization

(c) The atomizing medium like gas, liquid or combination of both

(d) Type of atomizer

(e) Characteristics of powder desired

On the basis of these variables efficiency of a particular design is decided. The overall efficiency is most conveniently related with the desired powder characteristics and the economy of production. Since number of variables are involved and all of them influence the design in different manner under the experimental conditions, it is very difficult to correlate them mathematically or emporically. Moreover the most efficient designs are patented and there is no scientific literature available for the desi-n of such nozzles.

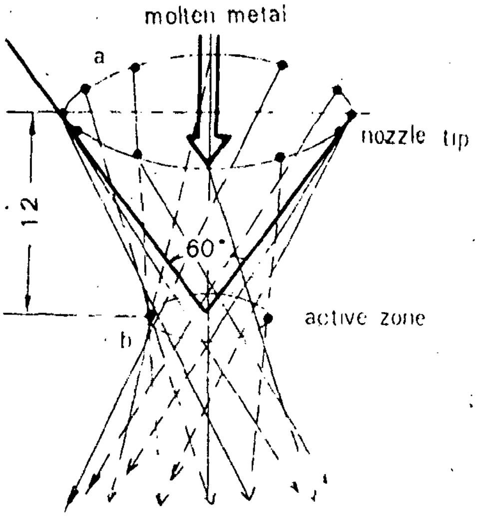


Fig 5 Diagrammatic sketch of spray form.

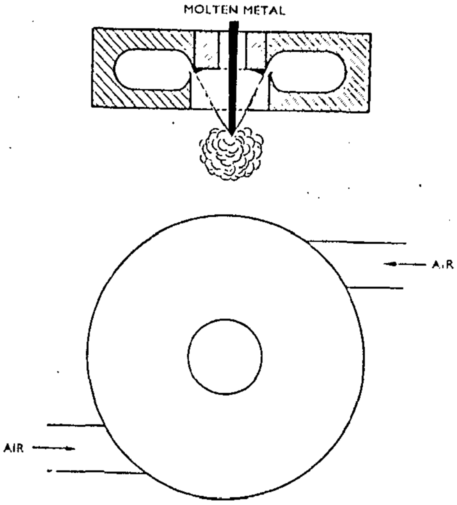
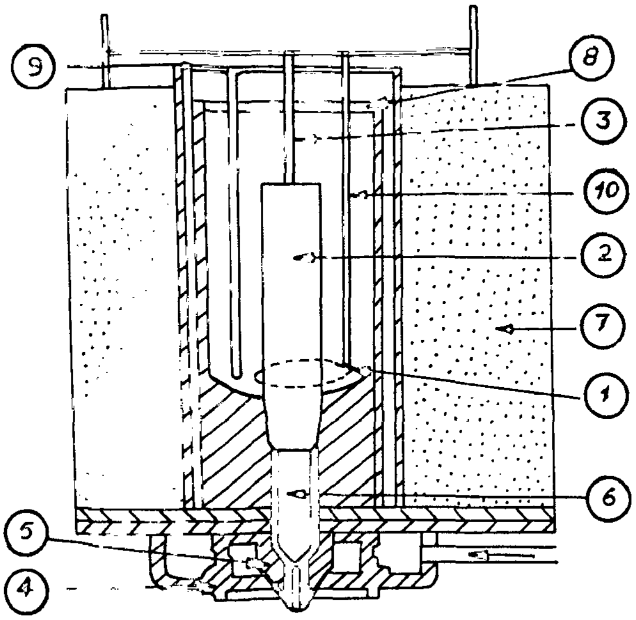


FIG. 6 Schematic representation of the Mannesmann process for atomizing liquid metals. (*Courtesy of the Institute of Metals*)



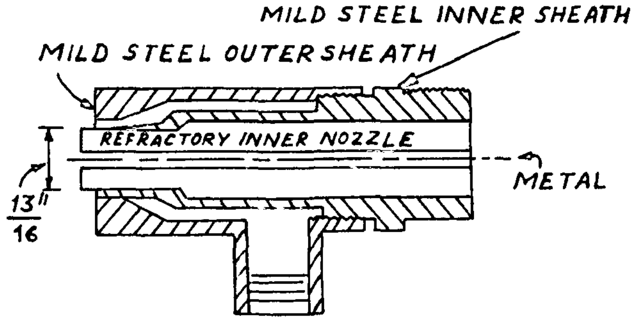


FIG. 7

FIG.8 CRUCIBLE & AIR NOZZLE ASSEMBLY USED FOR ATOMIZATION

1 GRAPHITE CRUCIBLE
2 GRAPHITE STOPPER
3 STAINLESS STEEL ROD
4. AIR NOZZLE ASSEMBLY
5. AIR NOZZLE ASSEMBLY
6. MILD STEEL NOZZLE (I.D. 283 MM)
7. RESISTANCE FURNACE
8. CRUCIBLE COVER
9. THERMOCOUPLE (SILICA TUBE ENCASED)
10. STAINLESS STEEL (INLET WITH STIRRER RING)

DOUBLE CHAMBER SPRAY

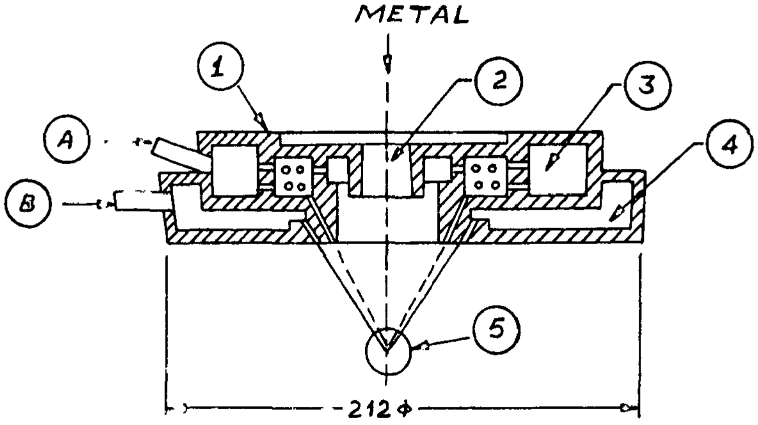


FIG. 9

1. SPRAY BODY 2. OVER FLOW DEVICE 3. OUTER GAS CHAMBER
4. CHAMBER FOR FEEDING LIQUIDS OR NATURAL GAS
5. SPRAYING ZONE
A. AIR FEED B. NATURAL GAS OR WATER FEED

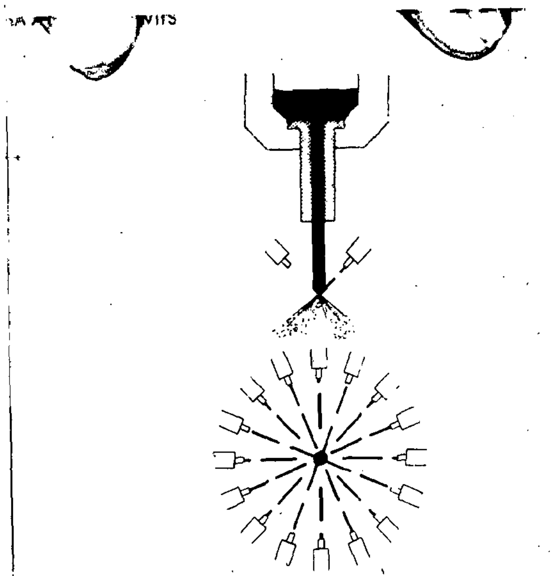


FIGURE 10
DIAGRAMMATIC SKETCH OF MULTIDIRECTIONAL HIGH SPEED DISCONTINUOUS COMPACT JET ATOMIZER ASSEMBLY

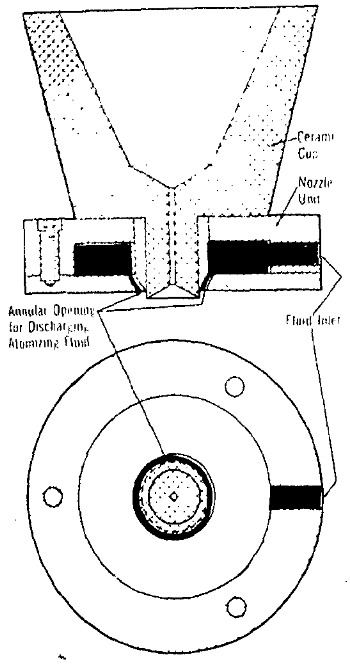


Fig. 11 — Sketch shows details of atomizing nozzle and pouring cup assembly. Molten metal is fed to the atomizing nozzle through a hole in the pouring cup tip. Hole size will vary, depending on the material to be atomized and the powder particle size desired.

Form of Gas Impingement

It could be visualized that when the air impinges only from one side of liquid stream, the main stream of air would not be utilized for secondary disintegration. If jet of air impinge from two or more sides then main stream of air would impinge to the liquid drop number of times, depending on number of sides of air jet applied. Thus for better disintegration the air should impinge in the form of a cone. Such types of nozzles have an hole at the centre to pass the liquid metal stream through vertex of cone of gas. For balancing the force of impingement following points are to be taken into consideration.

(1) There should be the same amount of air from all sides of liquid stream i.e. annular space should be of uniform thickness.

(2) Velocity from all sides should be same i.e. pressure should be uniform in the annulus.

Characteristically the spray formed by compressed gas shows a ruled surface as can be seen in Fig.(5). It will be noticed that the high velocity gas comes to focus which plays an important pole to disintegrate the molten metal streaming down from the metal nozzle, and then the gas diverges quite regularly from the focus. As the gas jet is led through two tangentially placed inlets, the gas stream is given a spiral motion which varies with the opening of annulus and, therefore it seems to act in different ways to the molten metal stream.

Thomson[1] had employed a nozzle for atomizing aluminium which has been shown in Fig. (7). During the atomization of aluminium, an operating temperature of about 800° C is generally employed and this imposes severe refractory corrosive condition on the nozzle material. Trials conducted with the common ferrous materials as construction material for the nozzle gave highly unsatisfactory results. The rapid formation of $FeAl_3$ caused irreparable deterioration of the nozzle. Even special heat resisting steel also failed within few minutes. Grophite, although giving a slightly longer life was never the less subject to a corrosive attack and was mechanically unsatisfactory. Eventually a special refractory material was tried and found to be eminently suitable. There appeared to be no corrosive attack and refractory strength was sufficiently high to withstand any normal treatment required during operation. The essential features of this particular type of nozzle are that the metal stream issuing from the central orifice is caught up by an annular blast of atomizing medium concentric with the jet, disintegrated and expelled from the nozzle as a confined spray of originally molten droplets which are chilled and solidify at some point distant from the nozzle. Details of nozzle design are shown in Fig. (7) and it will be noted that only the inner nozzle is constructed of refractory material, other parts not in contact with the aluminium being of mild steel.

For high melting point metals, nozzle used in Mannermann process[6] has been described - Schematic representation of which has been shown in Fig. (6). In this a stream of molten, low silicon, cast iron is atomized by a spiralling annular jet of compressed air, Oxidation of the particle surface occurs but with careful control of oxygen and carbon content a relatively pure iron powder can be produced by a heat treatment at 950° C. The process is still used for the production of iron powders but it can not be applied to alloys containing reactive metals, such as chromium, Aluminium and Titanium which form Stable Oxide

To get high purity powder via using less atomizing media of high velocity, a high speed discontinuous compact jet was designed by Domsa and Berborits[7] which is shown in Fig.(10). In practice, the number of jets used is limited on the basis of the design chosen. The orientation of the jet in the space, their speed, the frequency of pulses and their sequence in time are adjustable within wide limits.

Robest and Probot[8] have used a nozzle Fig. (11) for production of spherical copper powder. Accordingly their nozzle consists of insert body and plate. The insert is cylindrical ground with a 60° taper at one end which terminate in a rajor sharp edge. The body has an internal and external chamber. The external chamber receives the molten metal and feeds the internal chamber which is isolated by a gasket before the plate is placed in position.

Balasubramanian and Tendolkar[9] have used a atomizer which consists of an atomization chamber in the form of a truncated cone. At the top of this chamber an electric furnace is mounted. Air from the air chamber enters the air nozzle assembly, through 12 openings provided on its periphery. The upper piece flanged into the lower piece of the air nozzle directs the air through the annular openings Fig. (8).

Naida and Nichiporenko[12] have critically surveyed the basic design of sprays used for spraying liquid metals and an improved double chamber spray design is proposed, which substantially widens the engineering scope of the spraying process. One of the designs of double chamber spray has been shown in Fig. (9).

ATOMIZATION PROCESS

Mechanism of Atomization

Two process, namely disintegration and decreement of angularity of drops occur during atomization process as shown in Fig. (12).

Disintegration

Air impinges on liquid drop and results into disintegration of the drop. The disintegration may occur in many steps. Step is used as the maximum number of the times the disintegration of liquid drop occurs. These step depend on the following factors :

1. Velocity of air
2. Angle of nozzle
3. Over heating of liquid metal
4. Surface energy of liquid metal

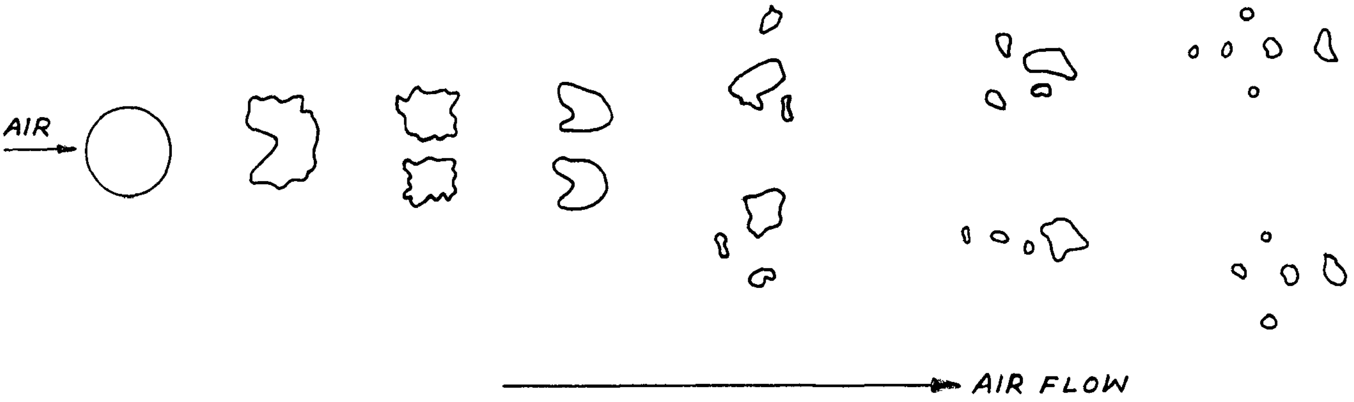
With increasing velocity air and over heating number of step increases. On the other hand surface energy of liquid and angle of nozzle decrease the number of step.

Decreement of angularity

After disintegration of liquid drop, the resulted drops have irregular shape and they have a tendency to become a spherical due to surface tension force of liquid. Attaining a spherical

MECHANISM OF ATOMIZATION

DISINTEGRATION PROCESS



DECREEMENT OF ANGULARITY

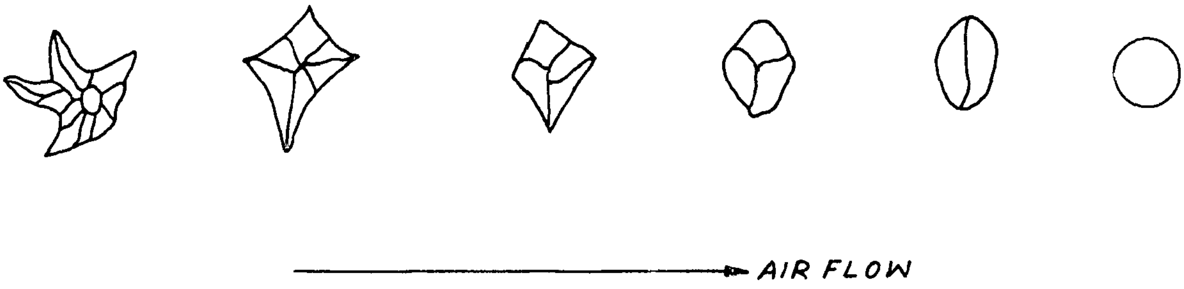


FIG. 12

shape of drop requires a minimum time which depend on intensity of surface tension. If drop solidifies before the minimum time, required to become a irregular drop to spherical drop, we got different shape of particle, depending on the time for which the disintegrated drop has been remained in liquid form. Attributing to the above fact, following variables may be considered as the most important variables for controlling the shape of atomized powder particles.

1. Surface tension of metal
2. Over heating of liquid metal
3. Heat transfer from liquid drop

With above consideration, shape and size of the atomized powder particle can be changed by following means :

1. Changing the kinetic energy of gas with
   - (a) Angle of impingement
   - (b) Amount of air flow
   - (c) Velocity of air
2. changing the solidification time of disintegrated drop. Following are the important considerations
   - (a) overheating of molten metal
   - (b) heat transfer from drop

(3) changing the surface characteristic on alloying or by any other method such as overheating etc.

Variables of atomization

Thomson[1] has studied experimentally some of the atomization variables. He concluded that control of the size characteristic is achieved by alteration of several process variables, the most important ones of which are, metal head, air pressure, metal temperature and metal orifice. In summary, the effects of these variables are.

Metal head :—

Slight increase in rate of atomization, corresponding decrease in fineness of product

Air pressure :—

Steady increase in rate of production, increase in fineness upto 50 p.si., thereafter a slight decrease. It may be due to the fact that pressure of air increases the impingement force as well as rate of flow of molten metal. First one is more effective upto 50 p.s.i. which results to fine powder, thereafter rate of flow is so pronounced which leads to coarser powder particle comparatively.

Metal temperature :—

Strongly marked linear decrease in rate of production, corresponding linear increase in fineness. By comparison with normal liquids it may be assumed that for liquid metals, over short range of temperature, both surface tension and viscosity are inversely proportion to temperature and therefore the resistance to disruption must also decrease with increase temperature. That is why fineness increase with temperature. Rate of production may decrease due to cubical expansion of metal on increasing temperature.

Area of Metal Orifice :——

Linear increase rate of production upto very high value (1200 lb/hr at 0.079 inch^2 area) corresponding linear decrease in fineness.

In practice alteration in these factors is employed to effect the major adjustment in size distribution which are required. Subsidiary variables such as inclination of external sheath effect such relatively small changes that they are unsatisfactory as a mean of process control, but may conveniently be employed in certain particular circumstances.

Size of atomized metal particles

Drop size distribution of atomized liquid metal, for production of metal powder is calculated. For reasons related to control and production efficiency in this field of powder metallurgy a knowledge of particle size distribution is of importance. In the course of research on the gas atomization of molten metal streams, model work is being carried out using low melting point alloys and tin to simulate the metal stream, with compressed air. Various workers have approached differently for estimation of drop size of atomized liquid. Their work may be summarised as follows.

Nubiyama[15] derived an equation for a specific design of converging pneumatic atomizer. The equation is widely quoted and contains the fluid properties mentioned above and a viscosity term but is only claimed to hold within the range of the physical properties of oil and a liquid. The mass mean particle dia d in micrometer is given by

$$X_m = \frac{585}{V}\sqrt{\frac{\gamma}{\rho}} - 597\left(\frac{\mu}{\gamma}\right)^{0.45} \times \left(\frac{1000\,Q_L}{Q_A}\right)^{1.5}$$

where V = relative velocity in m/sec of gas to liquid stream at impact derived from Anderson's curves[17] for velocity decay in sonic jets

γ = surface tension of liquid dynes/cm.

ρ = density of liquid gm/cc

μ = viscosity of liquid (poise)

Q_L, Q_A = volume flow rates of liquid air respectively

It may bo noted that for ratio of $\frac{\theta_A}{\theta_L} = 5000$, the second torm of the right hand side of equation contributes little to the predicted drop size in the case of inviscid fluids.

If the second term of the Nubiyama formula is neglected for the range of flow and inviscid fluids under consideration the ratio $\frac{1}{V}\sqrt{Y/\rho}$ will be seen to constitute the governing parameter, operation under the same conditions of gas pressure and liquid throughout, the ratio is clearly related to the weber number(w)

$$w = \frac{V^2 D}{r}$$ where D is diameter of liquid stream

which is a well known dimensionless number in the studies of liquid breakup phenomenon, representation the ratio of the fluid interfacial forces to the interfacial tension.

Wigg[10] obtained a correlation for waxes of various viscosities over a range of mass flow ratio. By the method of dimensional analysis. It is to be noted that he failed to achieve a direct correlation between wax and water. He believed this was due to coalescence of the water droplets. His formula for the wax gave the mass median diameter.

$$X_m = \frac{200 \times 10^3 \, Y_m^{0.5} \, M^{0.1} (1 + \frac{M}{A})^{1/2} \, D^{0.1} \, Y^{0.2}}{0.3 \, Y\rho}$$

where γ_m = Kinematic viscosity of liquid

M = Mass flow rate of liquid

A = Mass flow rate of gas

A study was done by H. Lubanstra[10] for correlation of spray ring data for gas atomization of liquid metals. The atomizing spray ring investigated consisted of a ring of discrete gas nozzles symmetrically surrounding the axis of a vertical liquid stream falling under gravity. He atomized iron by nitrogen . Relevant variables were used to study the variation of the geometric mean dia with the weber number (w). The ratio X_m/D was plotted initially against w, and some correlation was immediately apparent. However, improved correlation was obtained when account was taken of the mass flow ratio, total liquid to gas flow, M/A and the kinematic viscosity of the metal γm.

The data for the iron tests were plotted with all appropriate model spray data in the dimensionless form Hm/D verses ϕ and straight line was found out.

$$\phi = 10^7 \left[\left(1 + \frac{M}{A}\right) \frac{\gamma_M}{\gamma_g w} \right]^{0.5}$$

γ_g = Kinomatic viscosity of gas

and a simple relationship was developed

$$\frac{X_m}{D} = K \left[\frac{\gamma M}{\gamma g w} \left(1 + \frac{M}{A}\right) \right]^{1/2}$$

where K is a constant to be determined for particular conditions of spray ring and liquid stream. The value of K is seen to vary between 40 and 50 for the wide variety of spray ring condition investigated with the metal stream diameter varied from 1/4" - 7/8"

Purity of Atomized Powder

Producing metal powder by atomizing i.e. by breaking up a liquid stream of metal by a gas or liquid leads invitably to contamination of the powder. The principal contamination is usually oxygen. The degree of contamination depends mainly on the following factors.

(a) The chemical nature of the atomization agent and of the metal to be atomized

(b) The atomization temperature of the metal

(c) The specific Surface of the resulting powders

Some metals which are highly susceptible to oxidation can not be atomized with water or air because of the large amount of oxide formed. The oxides are difficult to reduce and in some instances e.g., Al and Mg. the sudden oxidation during atomizing may even lead to explosion.

The degree of oxidation of metal powder, which primarily depends upon the nature of the metal, during atomizing may be limited by using inert atomizing agents. In order to be completely inert, the agents must have very high purity. The use of such high purity inert agents on an industrial scale is not recommended, because of their high cost. In industrial practice, the atomizing agents contain certain chemically reactive impurities which lead to chemical and mechanical contamination of the powder. The degree of contamination is determined by the ratio of the amount of metal atomized to the amount of atomizing agent used as well as by the fineness of the particle. Since the amount of oxide contamination of the powder depends upon its specific surface it is evident that the oxide must be formed by a reaction at the particle surface. Therefore, the less atomizing agent is used for each kibogram of metal powder produced, the better will be the quantity of the metal powder. There is however, a minimum energy necessary for atomizing liquid metal. This means that the specific quantity of ggent used for atomizing can not be lowered below a certain limit. Domsa and Berkovits[7] have worked on how the highest energy of the atomizing stream can be combined with minimum quantity of atomizing medium using the nozzle.

Apart from using limited atomizing agents, other method is also used which is based on the principle of degree of oxidation of a particular element. For example, one of S and P in the molten iron makes S & P to oxidise rapidly during atomization and hence producing iron powder relatively purer. Accurately adjusting the amount of S or P in the melt it has been claimed to get very high purity iron powder. Etzel[18] has produced copper powder by atomization and suggested that the oxygen content of powders can be reduced by addition of alloying elements whose affinity for oxygen is greater than that of copper. After conducting lot of experiments he found that oxidation of the copper is completely inhibited by the presence of 0.3% P, which also results the minimum particle size.

Atomization of Aluminium - Tin Prealloyed

Atomized pure aluminium powder does not possess the required properties for use in the préparation of metallurgical products difficulties are encounteeed during pressing due to high plasticity, low apparent density and surfaće area and the poor flow characteristics of aluminium powder. Since aluminium is soft and malleable excessive cold welding occurs along the die walls. Since the surface is also comparatively small. There is not sufficient interlocking to give good cohessive strength, and hence the green strength of the compact is insufficient for further handling. In each cycle of operation some aluminium particles also stick to the die wall and high ejection pressures are required. This results in heavy wear of the die.

These difficulties could be remedied in aluminium tin powders made by atomization.

Tin imparts anticorrosion and antifrictional properties Particles of alloy size range nearly 74 microns showsatisfactory compressibility characteristics whatever the tin content of the alloy or the forming pressure. The higher is the green strength of the compacts obtained, with powders having tin content beyond 3 per cent, the ejection pressure is lower compared to powders with tin content less than 3 per cent.

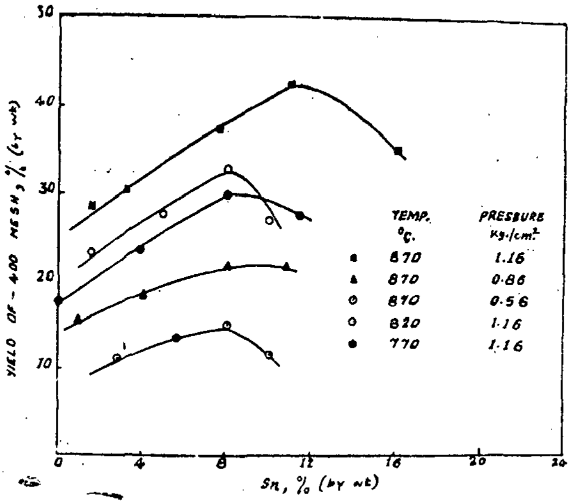


Fig.13—Effect of varying atomization pressure and temperature on atomizing efficiency for producing fine (—400

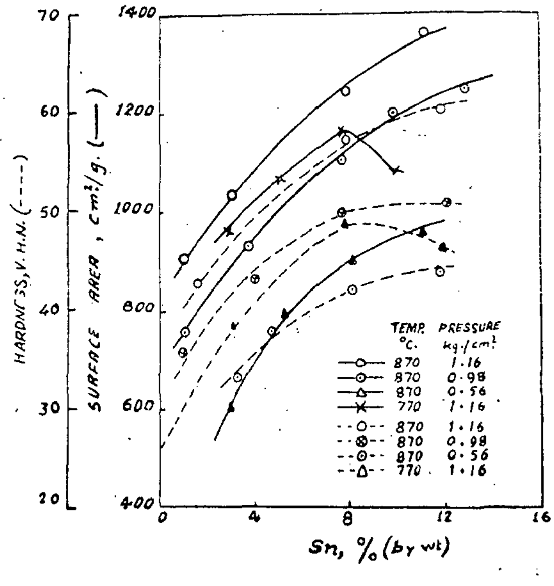


Fig.14 – Effect of varying atomization temperature and pressure on the surface area and hardness of alloy powders of different tin contents

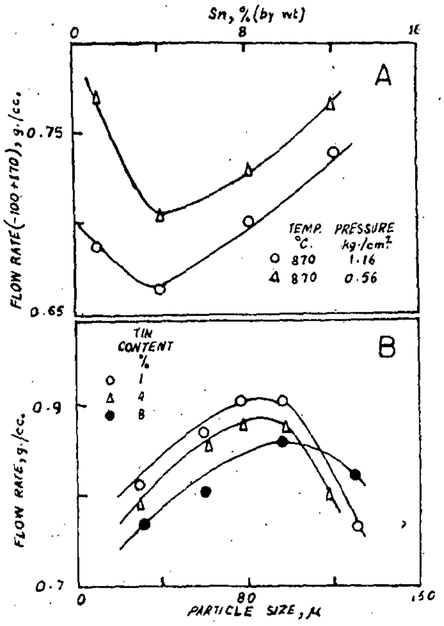


Fig.15— Variation of flow properties of powder produced under different conditions with respect to (A) particle size and (B) tin content

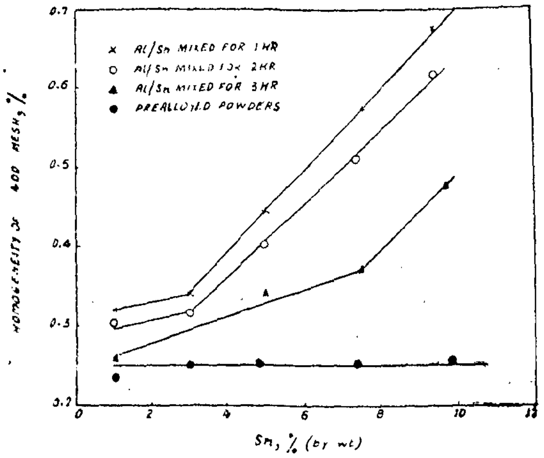


Fig.16 Homogeneity of alloy powders and elemental mixtures as measured by variation in tin content at different places in the powder mass

The low solid solubility of tin in aluminium makes the dispersion hardening effective even at higher operating temperatures. The rapid rate of cooling obtained in this process with tin content, helps in obtaining a product with high hardness, apparent density and surface area, but the efficiency of the atomizer is lower because of the agglomeration of the broken droplets.

In tin alloyed aluminium powder production, the composition of the powder changes with respect to particle size.

The effect of alloying additions is shown in the fig. (13) along with the effect of pressure and temperature on the atomization efficiency. As the amount of tin added is increased, the proportions of fine particles (-400 mesh) increases upto an optimum value. This is because tin requires lower disintegration energy. However, for higher percentage of tin, the rate of flow increases and hence more and more metal comes into contact with air at a particular instant and disintegration tends to be less complete. This effect counter balance the previous one, producing a peak corresponding to an optimum value of 8% tin at 0.56 kg/cm^2 pressure. For higher pressure even if more metal comes into contact with air, it gets disintegrated and hence the peak shifts toward the higher tin contents.

Surface area and hardness in Al - Sn prealloyed powder

It is evident that the hardness of the particles increases with increase in atomization temperature whatever the proportion of tin in the alloy may be. If a particular ligament cools or solidifies with a harder surface, the surface tension of the remaining uncooled liquid inside the core can not spheroidize the particles more effectively. Hence the ligament solidifies as such without decreasing the surface area.

Repeated experiments conducted at the department of metallurgical Engineering, Indian Institute of Technology, Bombay by M.S.N. Balasubramanian and G.S.Tendolkar[9] had shown that the surface area increases with increase in atomization temperature. Hence, atomization at a higher temperaturé yields particles with greater surface area. Also variation in surface area follows the same pattern as the variation in hardness with respect to tin content.

The rapid increase in hardness for initial increase in the percentage of tin (fig. 14) is due to the increasing close net work formation in the observed metallographic structure. The tin phase is very broadly dispersed in the aluminium. For higher percentage of tin, the amount of tin which can be dispersed in each individual particles increases, causing a corresponding increasing hardness.

Increasing the tin content beyond 8 per cent causes slight agglomeration of the dispersed phase and hence it may attain saturation state with 7 per cent tin. The possibility of agglomeration is more at lower agglomeration temperatures. Hence, actually a decrease in hardness for higher percentage of tin could be observed in their experiments. With higher atomization temperature and pressure the increase in the amount of oxide formed increase the hardness.

Flow properties of Al - Sn prealloyed powders

As the tin content is increased initially the flow rate decreases (i.e. the time taken for the flow increases). This is due to the rapid increase in the surface area of the particles. That is as the particles deviate more and more from spherical shape, it becomes difficult for them to roll round the flow meter. After this rapid increase in the surface area reaches a saturation limit and the increased hardness helps the particles to flow easily. Different flow characteristic at different atomization condition are represented in fig. 15. The Vanderwood forces at the surface of particles reduces the cold welding of individual particles during flow (i.e. the bridging effect is reduced).

The flow properties of powders of different sizes are presented in fig. (15). For all percentage of tin, a maximum flow rate is obtained (for a particle size of about 80 microns). The reason for an initial increase in the flow rate is the decrease in the nature of the shape factor. Below 80 microns, the interparticle friction and entrained gases decrease the flow rate.

Homogeneity of Al-Sn prealloyed powder

The β ray back scattering method was used by M.S.N. Balasubramanian and G.S. Tendolkar[9], to ascertain the homogeneity of the powders. Since the intensity of the scattered beam is a function of atomic number, different percentages of tin give different intensities. This scattered intensity is measured at different places in the powder mass and the difference in composity is calculated from the standard charts. Values of differences in composition at different places in the powder mass are shown in fig. (16). In this, result of similar analysis for elimental mixtures for different times of mixing - 400 mesh particles are also given. The maximum homogeneity of mixing can be had only for 400 mesh powders. Even for this an inhomogeneity of 0.7 per cent in 10 per cent tin is observed for 1 hr mixing. The alloy powders, however, show good homogeniety in all cases.

Apparent density of Al-Sn prealloyed powder

The usual range of apparent density of atomized aluminium powder is upto 0.95 gm/cc. The low apparent density requires long compression strokes and deep die, resulting in considerable loss of energy by way of friction and also excessive abrasion of the die walls. In the case of alloying with tin i.e. prealloyed powders, the apparent density increases upto 1.3 gm/cc and thus less difficulties in pressing are seen in prealloyed powders.

Sintering of atomized prealloyed powder

Sintering of Aluminium powder is very difficult because of the presence of oxide film on aluminium powder particles. This refractory oxide layer can be ruptured only at high compacting pressures, but then the porosity is drastically reduced. In porous self lubricating bearings, about 15 to 20 per cent porosity is essential and therefore, high compacting pressures are not permissible. To overcome the difficulties during sintering of Aluminium two methods namely the halide and hydride methods have been tried. In the former case, the aluminium powder is uniformly mixed with lead halide and sintered while in the latter case the sintering is carried out in an extremely dry hydrogen stream generated by the decomposition of a metallic hydride, to remove the diffusion inhibiting oxide film on aluminium powder particle surfaces. The main drawback with the halide method are that it requires very high compacting pressures and very long sintering time (upto 24 hrs.). The hydride method is too expensive because of the high cost of titanium hydride.

Agarwal and Ramkrishna[5] conducted certain experiment to investigate the aluminium base bearing materials. They have chosen tin as the alloying element because of its good anticorrosive and antifrictional properties. Further tin shows very

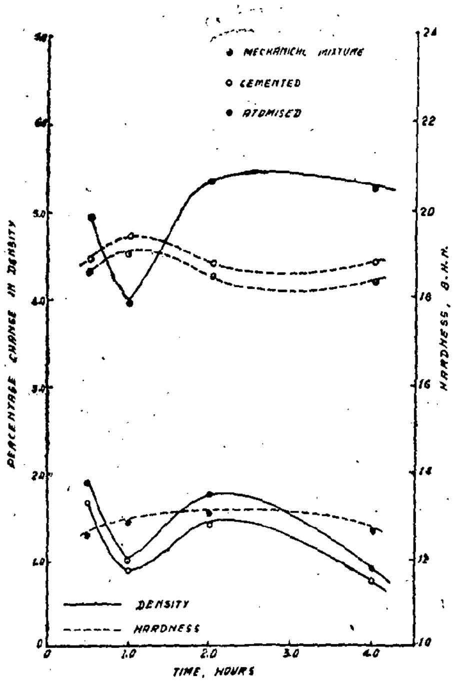


Fig. 17 Effect of sintering time on density and hardness for a constant sintering temperature, 600°C.

low solid solubility in aluminium, thus extending the use in higher operating temperatures and is capable of dispersion hardening. The low liquid temperature of the alloy makes the atomisation more easy and efficient. From preliminary studies, the useful range of tin content is found to be 3 to 8 per cent by weight. Aluminium tin alloy powders prepared by atomization and the results are compared with mechanically mixing the elemental powder. The general trend in the hardness is to decrease from half an hour to two hours and then to increase when sintered for four hours. But at 600°C (Fig. 17) the compacts made out of atomised powders give a maximum hardness for a sintering time of an hour. When the sintering temperature is increased, the hardness of the compacts decreases and density increases. But in the case of mechanically mixed powder, the density also decreases as the sintering temperature is increased.

In conclusion Agarwal and Ramakrishnan found that optimum value of sintering temperature and time for best properties are 600°C and 1 hour respectively. Considering the sintering atmosphere, vacuum sintering is found to give a superior product with better properties than the hydrogen sintering. Bearings made out of the atomised powders give much better properties than the mechanically mixed powders. Small addition of copper and nichel enhance the strength and other properties of bearings considerably.

CHAPTER - 3

EXPERIMENTAL SET UP

For producing metal powders, a atomizing unit (Fig.18) was installed, which consists of the following :

(1) Melting furnace

(2) Crucible with tube and heater

(3) Nozzle

(4) Air Compressor

(5) Stand on which furnace, nozzle and the powder collecting trough are mounted.

(1) Melting Furnace

Melting furnace was designed to get temperature upto 1000°C. Details of which are given below :-

Heating wire

Material	-	Manthal
Gauge	-	22 S.W.G.
Resistance	-	3.45 ohms/meter
Length	--	8.8 meter
Total resistance	-	30.36 ohms

ATOMIZER

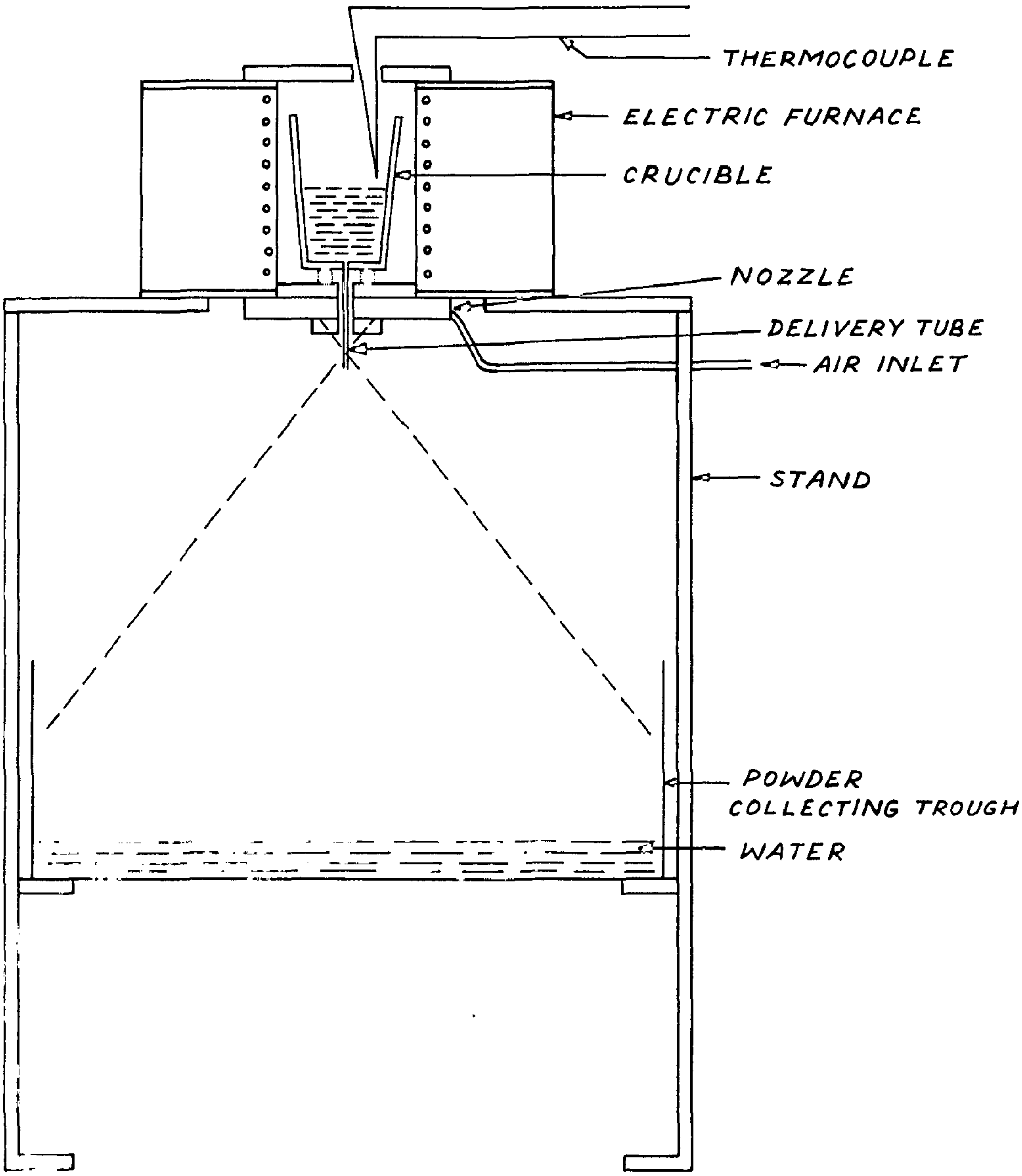
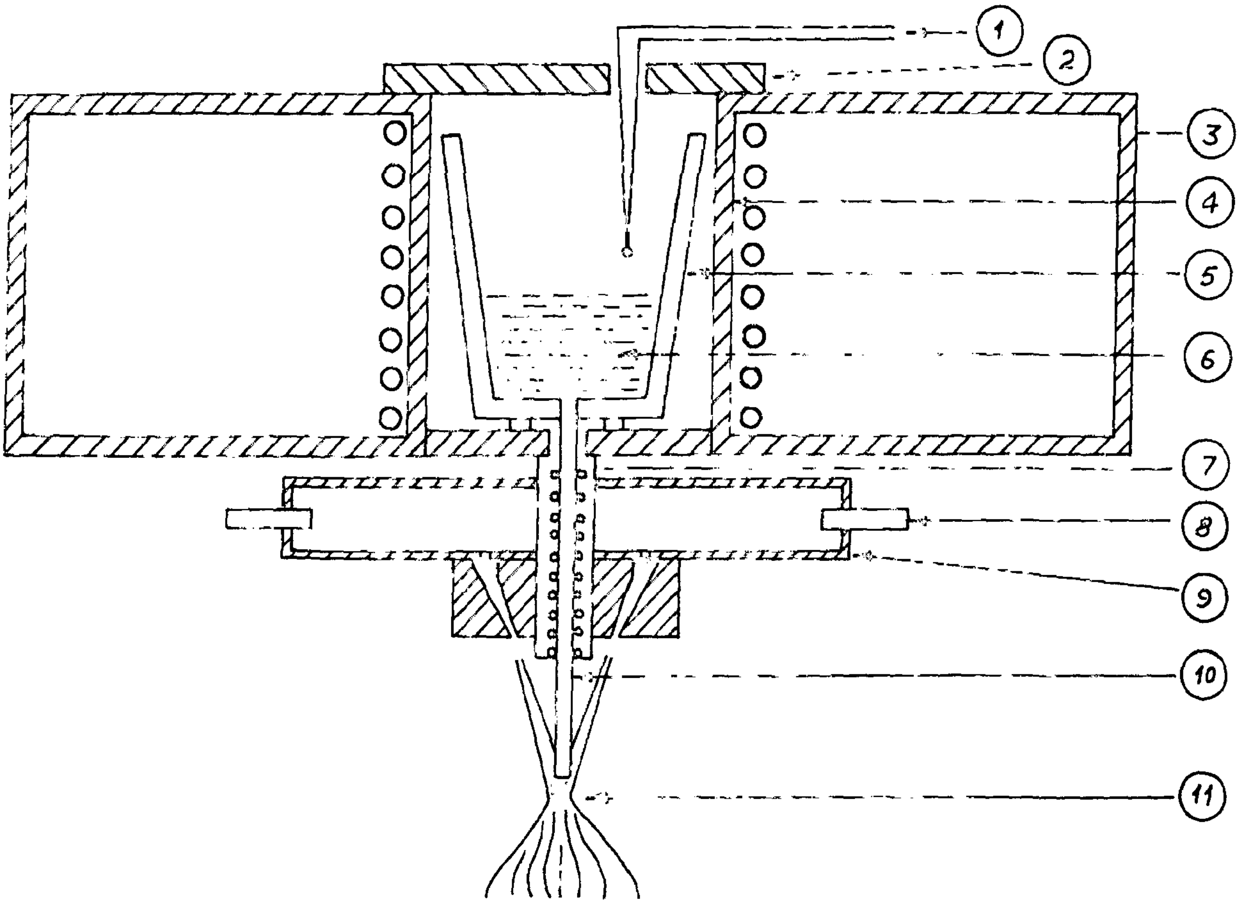


FIG. 18

FURNACE AND NOZZLE



1 THERMOCOUPLE
2 COVERING DISC
3 MELTING FURNACE
4 MUFFLE
5 GRAPHITE CRUCIBLE
6 METAL
7 HEATER
8 AIR INLET TUBE
9 NOZZLE
10 DELIVERY TUBE
11 ATOMIZING ZONE

FIG. 19

Fire clay muffle

Height - 15 cm
Cross section - 9 x 9 cm
Thickness - .5 cm.

Furnace rating

Voltage - 220 (max.)
Ampere - 7.25 (max.)
Watts - 1595

After the winding, a layer of fire creet about half centimeter thich is placed over the furnace winding. This layer of fire creet serves as insulating material and also gives sufficient stability and protection to the furnace winding. The asbestos powder is rammed around the crucible to give the furnace proper insulation.

(2) Crucible

Separate crucible is used for melting the metal and in the bottom of which fire clay tube is fixed to get the liquid metal in the form of stream. Tube dimensions have an important role during atomization which are as follows :

(a) Diameter - It relates directly the role of flow of metal i.e. rate of powder production and size of the powder produced. Small diameter of tube lead to fine size of the particle but flow in it will be difficult as capillary action will act and liquid metal may solidify in the tube due to more heat transfer from large surface area of well of tube. In case of small diameter tube, to compensate heat loses through wall of tube, a separate heater around the tube is provided to preheat the tube as shown in Fig. (19). Heater has the following data

Heating wire

Material - Kanthal

Gauge - 285 SWG

Resistance - 12.5 ohms/m.

Length - .85 m

Total resistance - 10.6 ohms

Heater rating

Voltage - 40 (max)

Ampere - 3.8 (Max.)

Watts - 152

To make the heater insulating from nozzle assembly. It was placed in the centre of an alumina tube with the help of asbestos rope.

(a) Length - Length of tube matters the proper flow of liquid metal at the time of atomization. Length of the tube may be such, that may open at or below or above the point of impingement of gas. If it opens above the point of impingement of gas then vacuum in the annular space around the tube will occur. Liquid metal try to fill this vacuum and as it comes in contact with metallic nozzle, it solidifies, results chocking of tube after some time. When tube opens at the point of impingement of air, due to more pressure of air as compared to the metal head pressure, air try to ascend in the tube, and will bubble through the liquid metal. For proper flow it is realized that tube should open below the point of impingement of gas.

Taking above considerations following tube dimensions were used

Length - 8 cm

Inner dia - .21 cm.

Thickness - .115 cm.

(c) Material of the Tube

The refractory tube should be non-corrosive to aluminium alloy and should have sufficient thermal shock resistance. For the purpose fire clay tube (used as insulating bits in thermocouple) was used.

(3) Atomizing Nozzle

Atomization method of powder requires to important forms of energy - thermal and kinetic, of which the later is provided with the help of a suitable nozzle. A nozzle is supposed to provide a high velocity cone of gas of uniform energy distribution which could be employed to disintegrate, in stages, the down flowing stream of liquid metal from furnace orifice.

As discussed earlier, the atomization will be most economical if impingement of gas will be equal from all sides of liquid stream. This can be obtained only if same amount of air with same velocity would impinge on liquid stream from all sides, hence uniformity in annualar space, pressure and symmetry with respect to liquid stream would have an important role in designing a nozzle. Following design aspects have been considered.

(a) Wind chart - For continuous flow of air with constant pressure, a wind chart is provided at the top of annulus. Size of wind chart may be as large as possible though it depends on annulus. The height of the wind chart is limited by a maximum permissible discent of metal stream before atomizing. Here it is taken approximately.

(b) Provision of two inlet - Placed symmetrical with respect to the annulus. These help to achieve an equitable distribution of pressure in the wind chest.

(c) Intermediary Channel - Connecting the wind chest and annulus this is in the form of a grooved cup which receives air from the wind chest and tend to equalize air pressure in the subsequent flow through the annulus.

(d) Connecting orifice - It transfer the air pressure from the wind chest to the intermediate channel. The suitable number and diameter of such orifice have been worked out from an estimation of working pressure range and the size of the wind chest.

(e) Projected periphery - It was provided at the internal solid metallic part (C), as can be seen in fig. (20), for the purpose of getting uniform annulus and symmetry with respect to liquid stream.

(f) Cone Angle - It would determine the following variables of atomization.

(a) Area of effective disintegration. Greater this magnitude of area, smaller will be the specific energy per unit area available for disintegration, and hence smaller the rate of atomization.

(b) Effective component of gas energy used up in atomizing. This component goes on decreasing as the cone angle is increased.

(c) Distance of point of application of compressed air from the point of emergence from the nozzle. This factor would act in a counter direction to factor (b) due to the direct "dissipation" effect caused by the air pressure.

(g) Annular Space - It is provided for air flow before striking the metal stream. The consideration for keeping an optimum uniform space arises from the importance of obtaining a symmetrical concentric jet about the axis of down coming stream. An unsymmetrical jet may cause many diversities, predominantly, a wide size distribution range and nonuniform particle shape. Precise estimations in this regard have been made possible by experience.

(h) Material Consideration - The most important requirement from the material of application is its machinability. The other property desirable from the material under consideration is its fabricability which includes weldability. Mild steel in various forms viz., sheet, block etc., fulfils all these requirements.

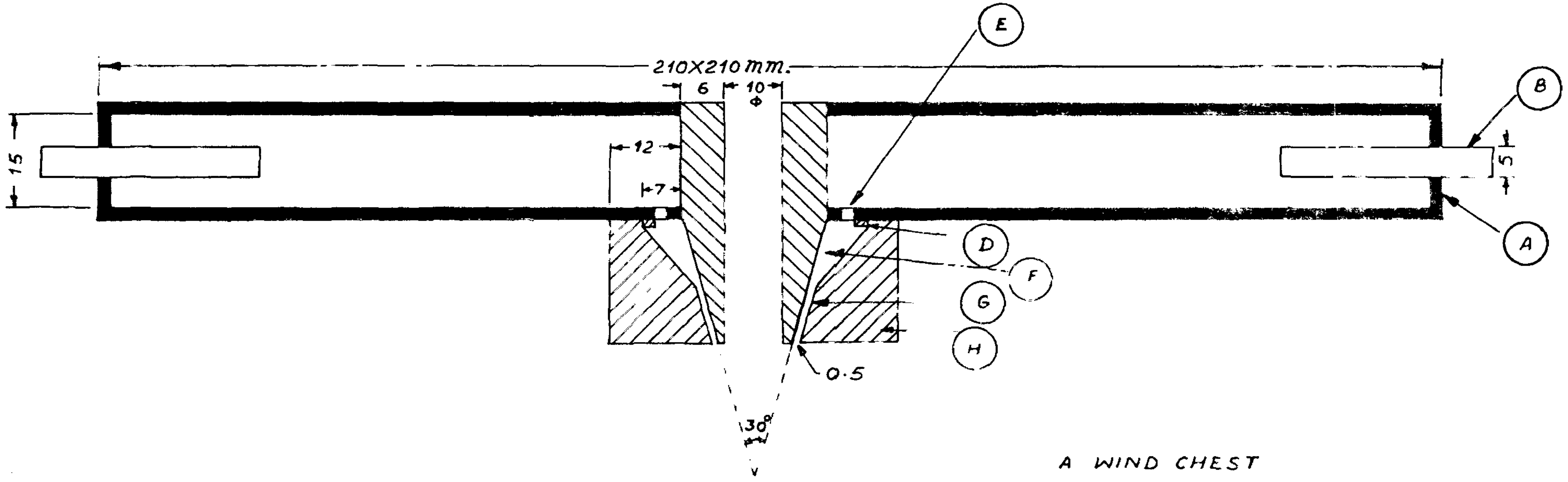
(i) Fabrication of Nozzle - Nozzle was fabricated in the following three parts.

Wind Chest - It was made with the mild steel sheet of 3 mm thickness by welding it, according to the dimension shown in fig. (20).

Internal Solid Part (C) - which has hole at the centre to pass liquid stream, projected periphery to fit H part and tapered edge to make angle of cone. It was fabricated on the lathe from the solid block of mild steel.

Outer Hollow Part (H) - It was fitted on internal solid C part to make intermediate channel and angle of cone of air. It was also fabricated on the lathe from the solid block of mild steel. These three parts are welded together to form the nozzle as shown in Fig.(20).

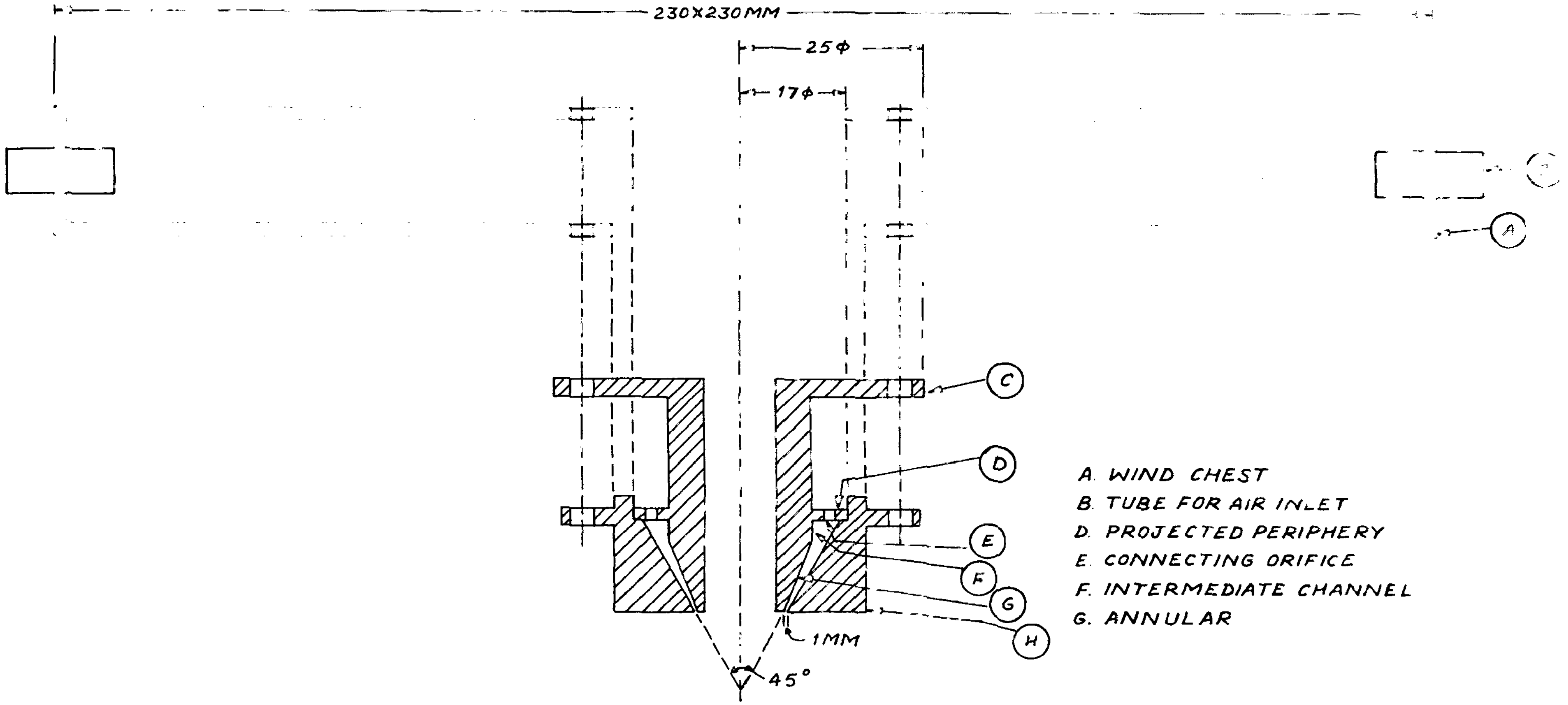
NOZZLE 1



A WIND CHEST
B TUBE FOR AIR INLET
D PROJECTED PERIPHARY
E CONNECTING ORIFICE
F INTERMEDIARY CHANNEL
G ANNULUS

FIG. 20

NOZZLE



A. WIND CHEST
B. TUBE FOR AIR INLET
D. PROJECTED PERIPHERY
E. CONNECTING ORIFICE
F. INTERMEDIATE CHANNEL
G. ANNULAR

FIG. 21

Drawback - During welding some deformation occurs which results nonuniformity in annular space.

Improved design shown in Fig. (21) was tried where whole welding is replaced by nut and bolt system perfect symmetry and air tightness was obtained.

(4) Air Compressor

The air compressor assembly can generate a pressure upto 110 psi. Due to its low capacity and low rate of generation of air pressure, this maximum pressure decreases to 50 psi within 2 minutes during working. This caused a limitation in studying atomizing conditions fully. A three way channel is provided to divert the blown air into two inlets of the nozzle. The pipe fittings consist of pressure rubber tubings capable of withstanding the normal pressure.

(5) Stand for mounting furnace, nozzle and collecting Trough

It was made of slotted angle iron in which a provision was made for mounting the furnace and nozzle. A shifting platform for stationing the powder-collecting trough was provided below the atomizing assembly. The trough was made out of the tin sheet which can be introduced in and pushed out of its stationing platform. It has been shown in Fig. (19).

CHAPTER - 4

EXPERIMENTAL PROCEDURE

Experiment consists of production of aluminium and its alloy powders and study of their characteristics. Different steps involved in the experiment are as follows:

1. Adjustment of different controls of atomizer
2. Melting of metal
3. Formation of metal Stream
4. Blowing the compressed air
5. Ceasing of metal flow
6. Collection of powder
7. Sieve analysis
8. Shape study
9. Sinterability Study

1. Controls of different parts of atomizer

First step is to fix the nozzle on the atomizer stand followed by the placement of the furnace in such a manner that the orifice of the furnace and the nozzle opening are concentric. Inside the furnace, crucible with delivery tube is placed as shown in Fig. (19). The tub for collecting powder is cleaned and placed at the projected angle iron sleves provided below the nozzle assembly in the atomizer. This tub is then filled with water to a height of about 4".

After this, place the air compressor at one side of atomizer and the temperature control unit on the other or as per convenience of working.

2. Melting of Metal

Make the furnace switch on after connecting it to energy regulator and latter to mains. Temperature is indicated with the help of Pt & Pt-Rh thermocouple, connecting to the temperature indicator with the help of compensating leads.

A weighed charge of metal to be atomized is placed inside the furnace from the top and the furnace mouth is covered, and the metal is allowed to melt and heated to the desired temperature.

3. Formation of Metal Stream

Switch on the heater around delivery tube after connecting it to main through transformer. Gradually voltage is increased from 0 to 35 volts. After some time, metal will continue to flow through delivery tube. Make the supply voltage 20.

4. Blowing the compressed air

After formation of stable liquid stream, valve of the compressor, maintained at 110 psi is opened to blow the compressed air through the nozzle. When pressure drops to 50 psi, valve of the compressor is turned to stop air blowing.

5. Ceasing of metal flowing

Switch off the heater around the delivery tube and metal flow is ceased by cooling the delivery tube by touching it with a steel bar.

6. Collection of powder

The water tub is withdrawn from the atomizer and water is removed from it. The remaining water alongwith the powder produced is transferred in another small vessel or on a paper and is dried in atmosphere. The water tub is now ready for further atomization . Dry powder is collected and kept in polythene bags.

7. Sieve Analysis

A dried 80 gm powder sample is placed on top of a series of sieves and is shaken for 20 minutes with the help of sieve shaker. The portion of the powder retained on each sieve is collected and weighed and expressed as percentage of total powder retained over a particular screen.

8. Shape Study

Shape of various powder is studied with the help of Stereomicroscope by sprinkling the powder on glass plate. Magnification used for studying the shape was 40 X.

9. Sinterability Study

Powders of particular size distribution is prepared by mixing the sieved powders. This powder, with 3% Stearic acid and methyl alcohol is ground with the help of pestle and mortar. Compacted specimens are prepared with the help of punch and die on the universal testing machine. Accurate dimensions of green compacts are measured with screw gauge.

Compacted specimen are sintered in a vacuum of order of 5 to 7 microns with vacuum sintering assembly which consists of vacuum unit i.e. diffusion vacuum pump joined with rotary vacuum pump and sintering furnace compacted specimen are presintered for degasing at 250°C for 1/2 an hour and finally they are sintered at 600°C for different time intervals.

Accurate dimension of sintered specimen are measured with screw gauge and change in density is calculated.

For hardness measurement, sintered specimens are ground on emery paper upto three zero number and BHN value is tested using 2.5 mm steel ball and 15.6 kg. load on Brinell hardness tester.

Curves for showing change in density and hardness value with sintering time are plotted.

CHAPTER - 5

RESULTS & DISCUSSIONS

Size Analysis of the Powder

Following variables are kept constant during atomization of aluminium alloy powders

1. Metal Temperature	... 800°C		4. Annulus	... 2 mm
2. Air pressure	... 110 - 50 psi		5. Orifice dia.	... 2.1 mm
3. Nozzle angle	... 30°			

Table - 1 Size analysis of atomized Aluminium Tin powders

Sieve No.	Sieve opening microms	Aluminium		Al-2% Sn alloy		Al-3% Sn alloy		Al-5%Sn alloy	
		% over size	Cumulative % oversize	% over size	Cumulative % oversize	% over size	Cumulative % oversize	% over size	Cumulative % oversize
70	208	68.8	68.8	55.7	55.7	54.1	54.1	50.1	50.1
100	150	8.7	77.5	13.4	69.1	13.6	67.7	15.3	65.4
120	130	8.7	86.2	10.6	79.7	11.6	79.3	12.7	78.1
150	105	3.2	89.4	5.5	85.2	5.2	84.5	5.7	83.8
170	90	1.0	90.4	0.8	86.0	0.8	85.3	1.1	84.9
200	75	-	90.4	-	86.0	-	85.3	-	84.9
240	63	6.0	96.4	9.1	95.1	10.2	95.5	10.2	95.1
300	53	0.4	96.8	0.7	95.8	0.4	95.9	0.3	95.4
Pan	-	3.2	100.0	4.2	100.0	4.1	100.0	4.6	100.0

Table 2 - Size analysis of Aluminium - Copper Powder

Sieve No.	Sieve opening No.	Pure Aluminium		Al-2% Cu alloy		Al-4% Cu alloy	
		% over size	Cumulative % oversize	% oversize	Cumulative % oversize	% oversize	Cumulative % oversize
70	208	68.8	68.8	61.9	61.9	60	60
100	150	8.7	77.5	12.0	73.9	11.8	71.8
150	105	11.9	89.4	15.2	89.1	15.2	87.0
170	90	10.0	90.4	0.3	89.4	-	87.0
200	75	0.0	90.4	-	89.4	-	87.0
240	63	6.0	96.4	7.1	96.5	8.3	95.3
300	53	0.4	96.8	0.4	96.9	0.5	95.8
pan	-	3.2	100.0	3.1	100.0	4.2	100.0

Table 3.- Size analysis of Aluminium alloy powders

SERIAL NO.	% Tin	Percentage of - 100 mesh	% Copper	Percentage of - 100 mesh
1	0	22.5	0	22.5
2	2	30.9	2.0	26.1
3	3	32.3	4.0	28.2
4	5	34.6		

Sinterability of Powders

The powder for sintering study was prepared by mixing the sieved powder in the following proportions :

Table 4 - Size distribution of powder for compaction

Mesh No.	Percentage
- 70 + 100	10
- 100 + 120	10
- 120 + 150	10
- 150 + 170	5
- 170 + 240	5.5
- 240 + 300	5
- 300	5

Powders were ground with the help of pestle and mortar with stearic acid and in presence of methyl alcohol for half an hour. Compaction was made with the help of punch and die on universal testing machine.

Constant variable used in sintering was as follows :

Compaction pressure	...	2500 kg/cm^2
Sintering temperature	...	600°C
Sintering atmosphere	...	Vacuum of the order of 5-7 microns

Table 5 - Sinterability of aluminium and its alloy powders

S.No	Metal powder	Before sintering		Sintering time	After sintering			Change in density %age	Hardness BHN
		Dia. cm	Height cm		Dia cm.	Height cm	Mass gm		
1	2	3	4	5	6	7	8	9	10
1	Al powder (grinding)	1.008	0.387	1/2	1.009	0.338	0.737	- 0.42	7.1
		1.008	0.381	1	1.008	0.380	0.735	+ 0.28	16.1
		1.008	0 359	2	1.008	0.358	0.682	+ 0.28	12.4
		1.008	0 418	4	1.008	0.417	0.791	+ 0.37	15.6
2	Al powder (Nongrinding)	1.009	0.458	1/2	1.011	0.459	0.900	- 0.57	10.8
		1.009	0.516	1	1.009	0.515	0.996	- 0.21	15.9
		1.009	0.388	2	1.010	0.394	0.733	- 1.16	11.3
		1.012	0.396	4	1.011	0.397	0.765	- 0.66	16.2

Table 5(Contd.)

1	2	3	4	5	6	7	8	9	10
		1.011	0.283	1/2	1.010	0.283	0.538	+ 0.21	8.9
		1.011	0.351	1	1.009	0.350	0.671	+ 0.71	14.5
3	Al-2 % Cu powder	1.011	0.378	2	1.008	0.377	0.735	+ 0.87	21.2
		1,011	0.448	4	1.008	0.445	0.841	+ 1.28	24.60
		1.011	0.448	8	1.008	0.445	0.841	+ 1.28	20.8
		1.010	0.425	1/2	1.010	0.425	0.840	0	10.20
		1.010	0.422	1	1.010	0.423	0.832	- 0.21	14.1
4	Al powder and 2% Cu powder	1.009	0.352	2	1.009	0.353	0.682	- 0.3	13.5
		1.010	0.379	4	1.010	0.378	0.812	+ 0.3	13.9

Table 5(Contd.)

1	2	3	4	5	6	7	8	9	10
5	Al-2% Sn powder	1.008	0.404	1/2	1.009	413	0.783	- 2.38	14.2
		1.008	0.442	1	1.009	444	0.831	- 0.68	16.7
		1.008	0.451	2	1.008	453	0.875	- 0.49	17.3
		1.008	0.349	4	1.008	351	0.676	- 0.55	16.3
6	Al-3% Sn powder	1.008	0.284	1/2	1.012	0.288	0.542	- 2.18	13.2
		1.008	0.333	1	1.008	0.333	0.643	0	16.2
		1.008	0.369	2	1.008	0.369	0.715	0	14.8
		1.008	0.417	4	1.008	0.415	0.793	+ 0.5	16.8

Table 5(Contd.)

1	2	3	4	5	6	7	8	9	10
	~~1.011~~	~~0.265~~ 1.011	0.265	1/2	1.019	0.274	0.515	- 4.79	13.0
		1.011	0.327	1	1.013	0.328	0.651	- 0.91	13.6
7	Al-5% Sn powder	1.011	0.319	2	1.013	0.322	0.524	- 1.49	12.6
		1.011	0.362	4	1.011	0.362	0.681	0	13.2
		1.013	0.496	1/2	1.014	0.495	0.980	0	13.2
		1.013	0.408	1	1.013	0.407	0.822	+ 0.12	12.5
8	Al powder and 2% Sn powder	1.013	0.415	2	1.013	0.419	0.838	- 0.92	11.9
		1.013	0.448	4	1.014	0.449	0.891	- 0.45	13.4

Table 5(Contd.)

1	2	3	4	5	6	7	8	9	10
		1.012	0.352	1/2	1012	0.362	0.712	- 2.78	8.8
		1.012	0.336	1	1014	0.343	0.678	- 3.39	14.5
9	Al powder, 2% sn powder and 1% Cu powder	1.012	0.388	2	1013	0.396	0.770	- 2.39	12.7
		1.012	0.357	4	1016	0.363	0.736	- 2.42	14.8
		1.012	0.357	8	1015	0.363	0.736	- 2.39	14.3

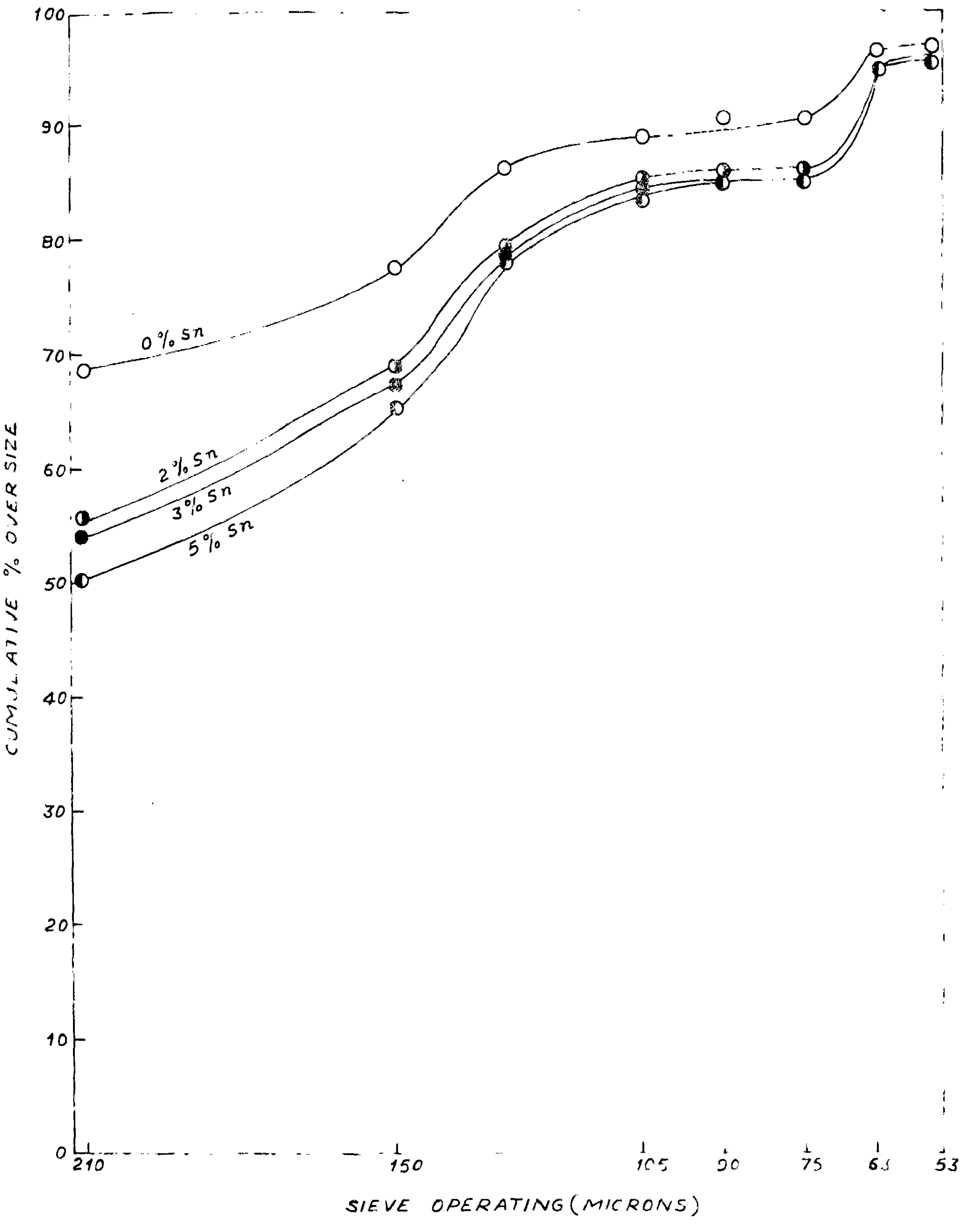


FIG.22 SIEVE ANALYSIS OF ALUMINIUM TIN POWDERS

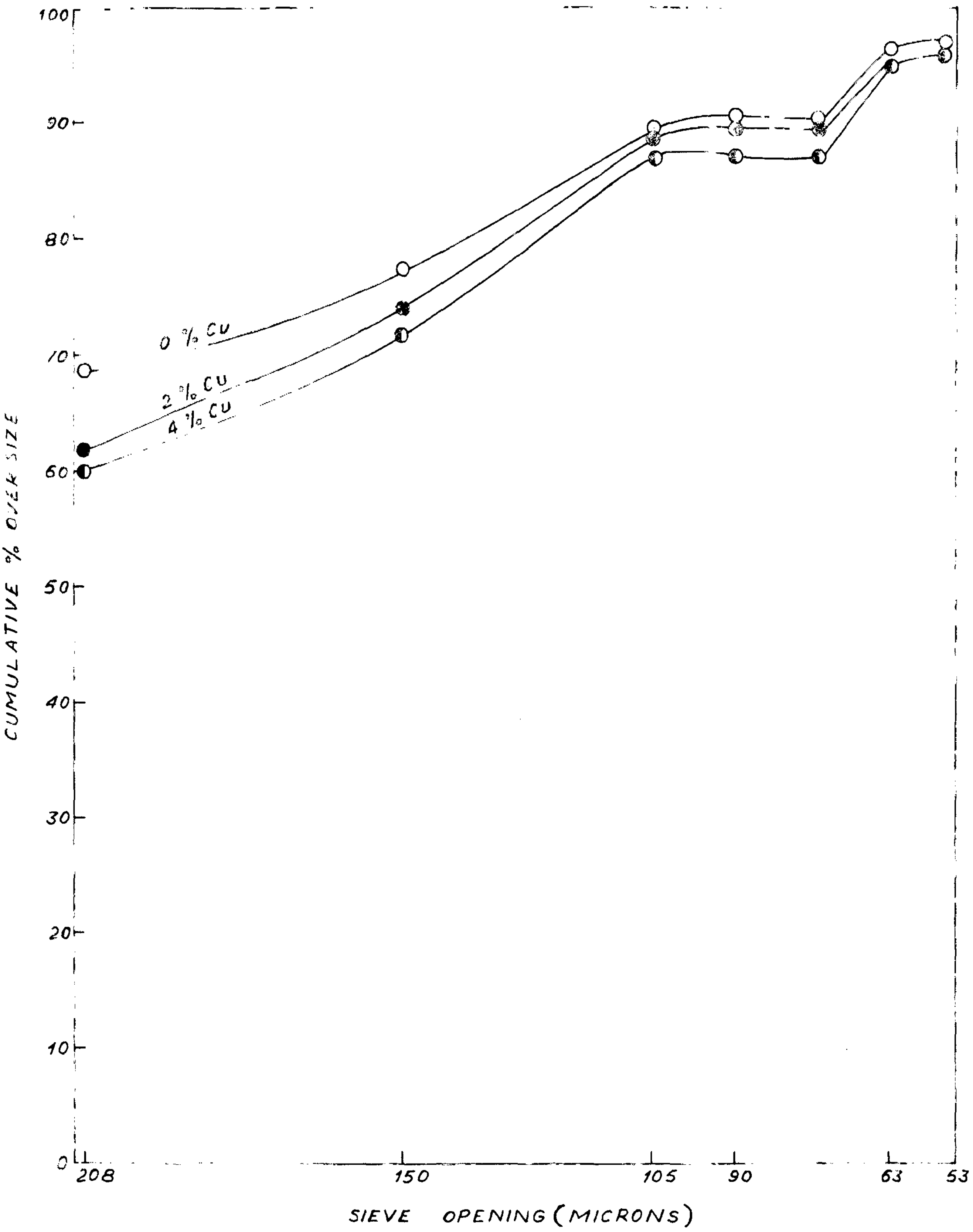


FIG. 23 SIEVE ANALYSIS OF ALUMINIUM COPPER POWDERS.

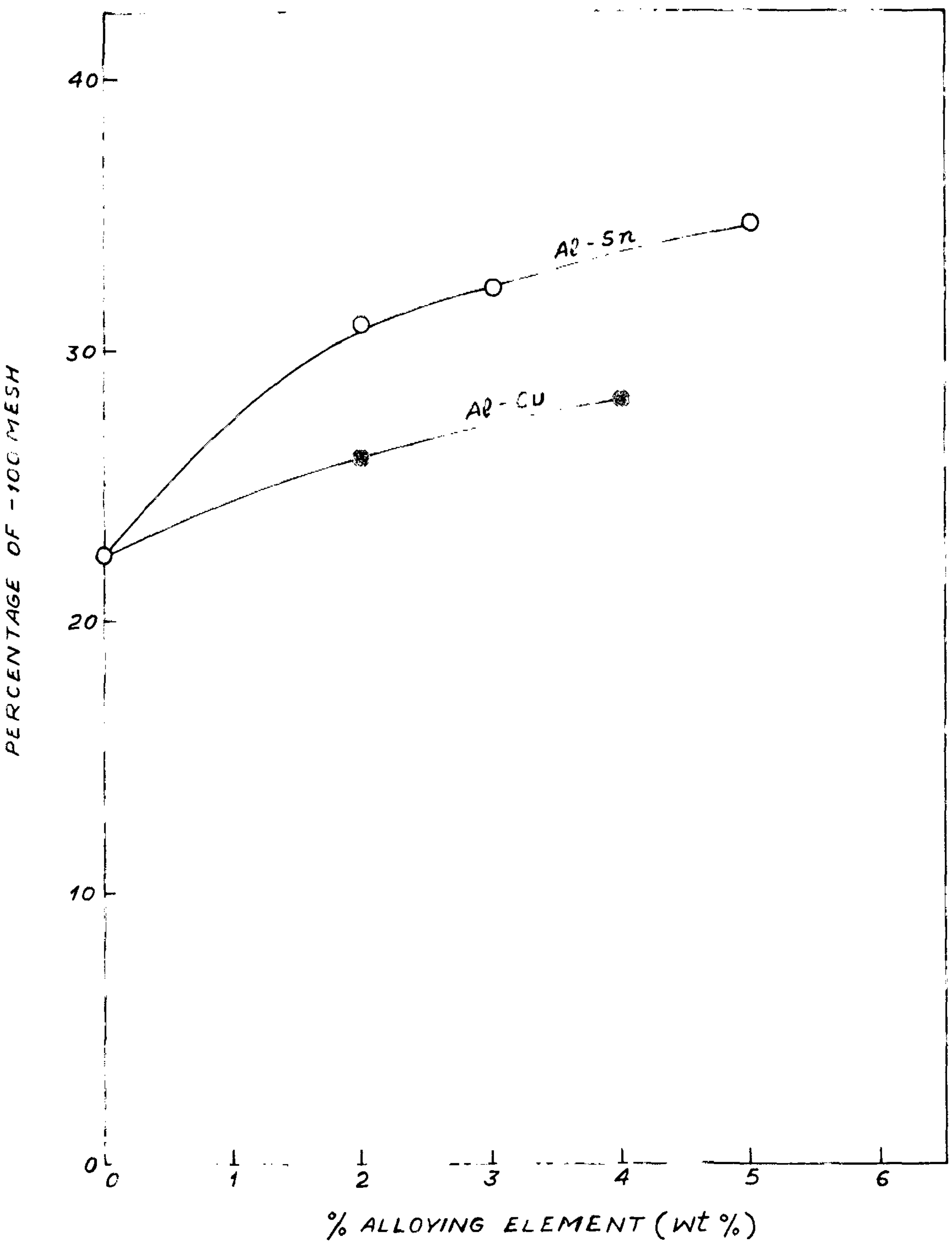


FIG 24 SIEVE ANALYSIS OF ALUMINIUM ALLOY POWDERS.

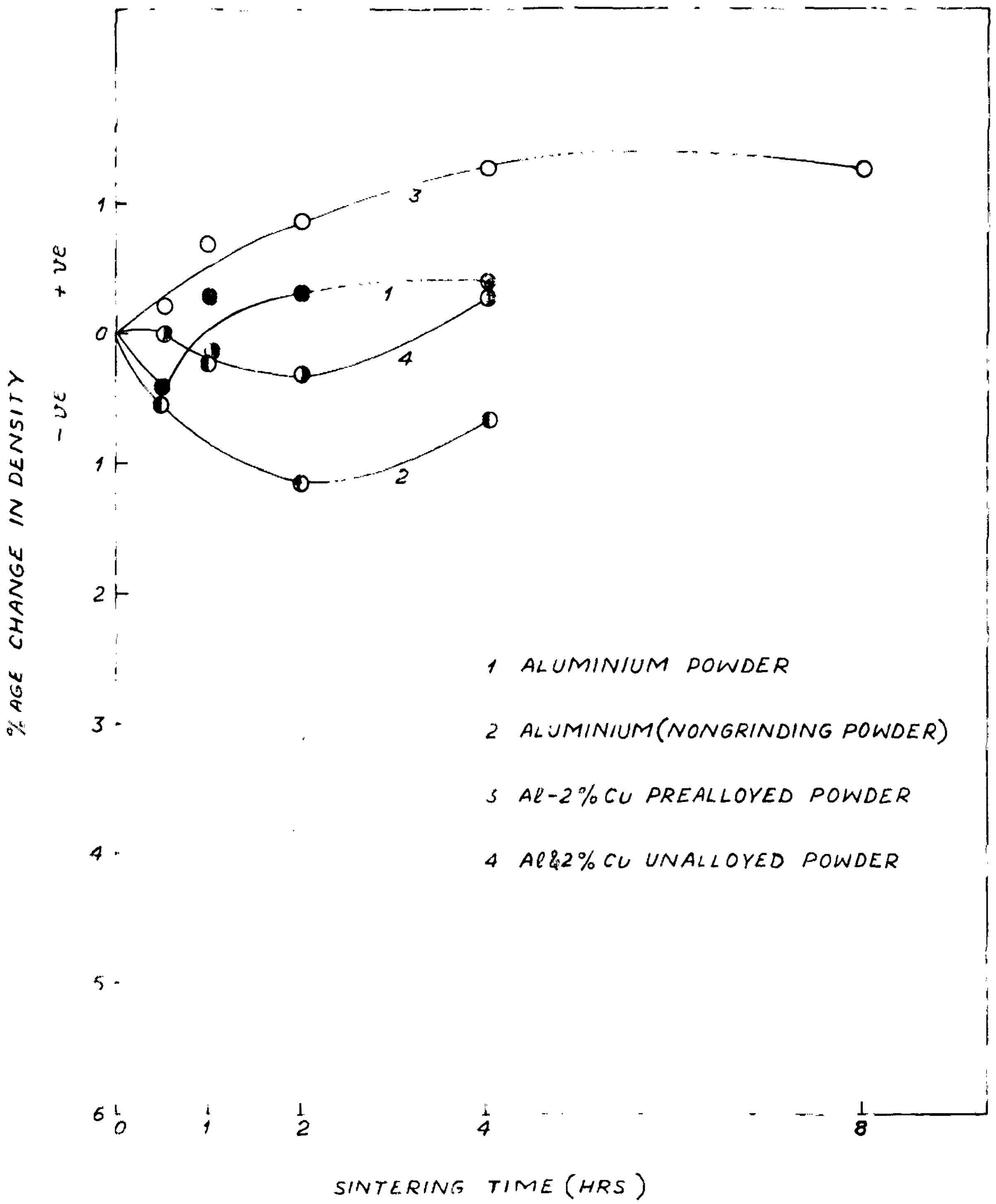


FIG 25 DENSITY CHANGE OF ALUMINIUM AND ITS ALLOY POWDERS DURING SINTERING AT 600°C

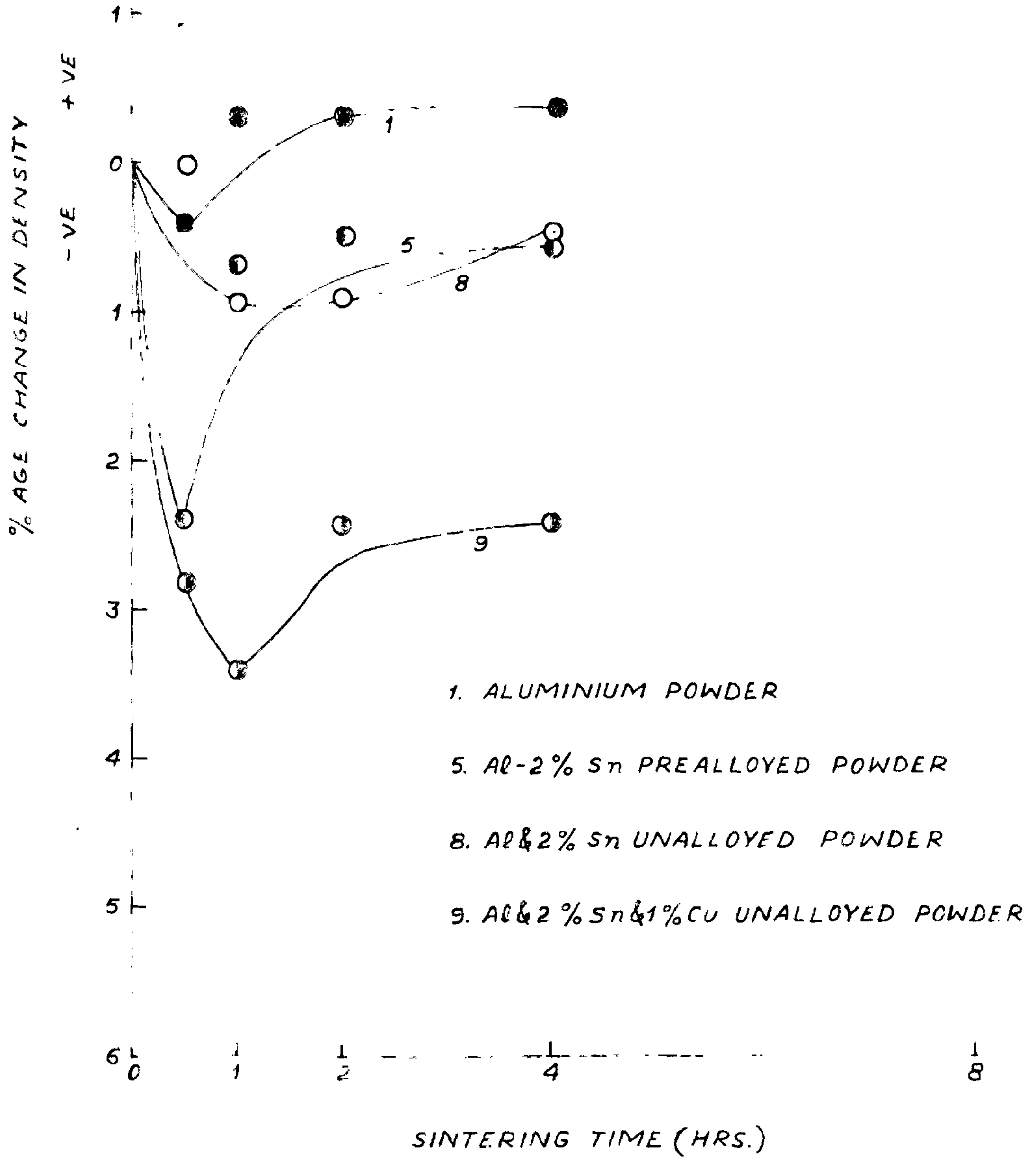


FIG. 26 DENSITY CHANGE OF ALUMINIUM AND ITS ALLOY POWDERS DURING SINTERING AT 600°C

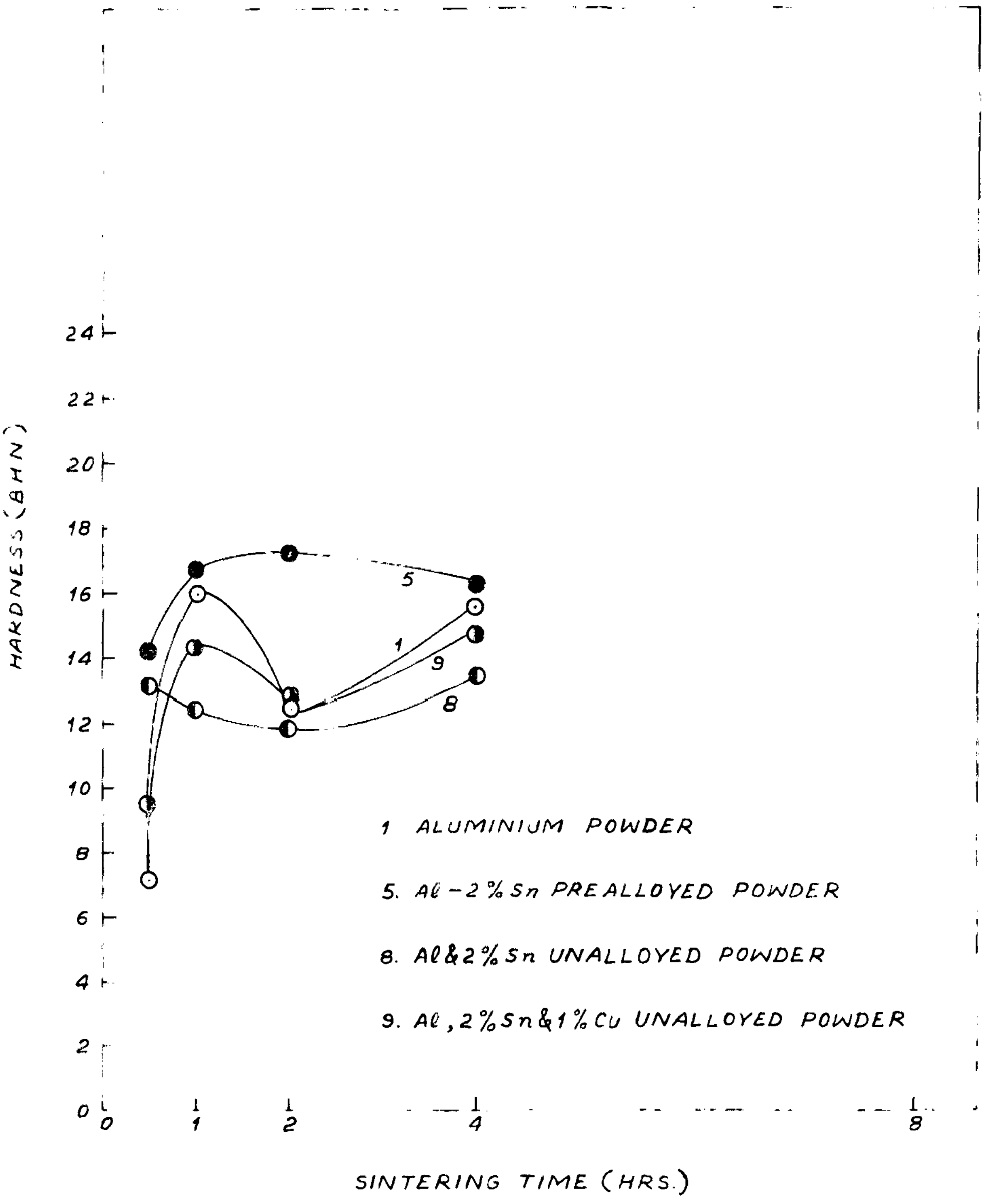


FIG. 27 HARDNESS OF ALUMINIUM AND ITS ALLOY POWDER DURING SINTERING AT 600 °C

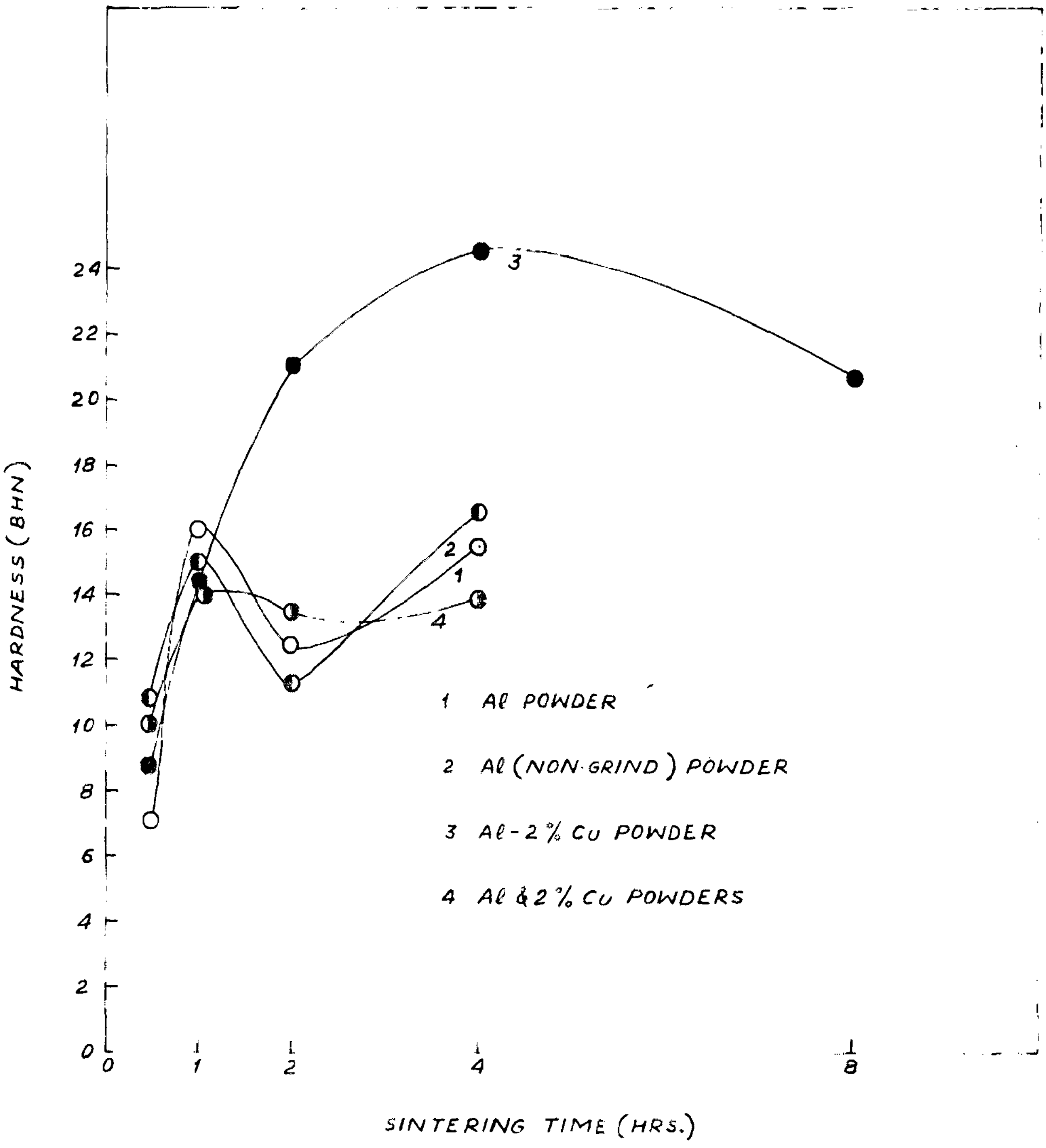


FIG.28 HARDNESS OF ALUMINIUM AND ITS ALLOY POWDER DURING SINTERING AT 600°C

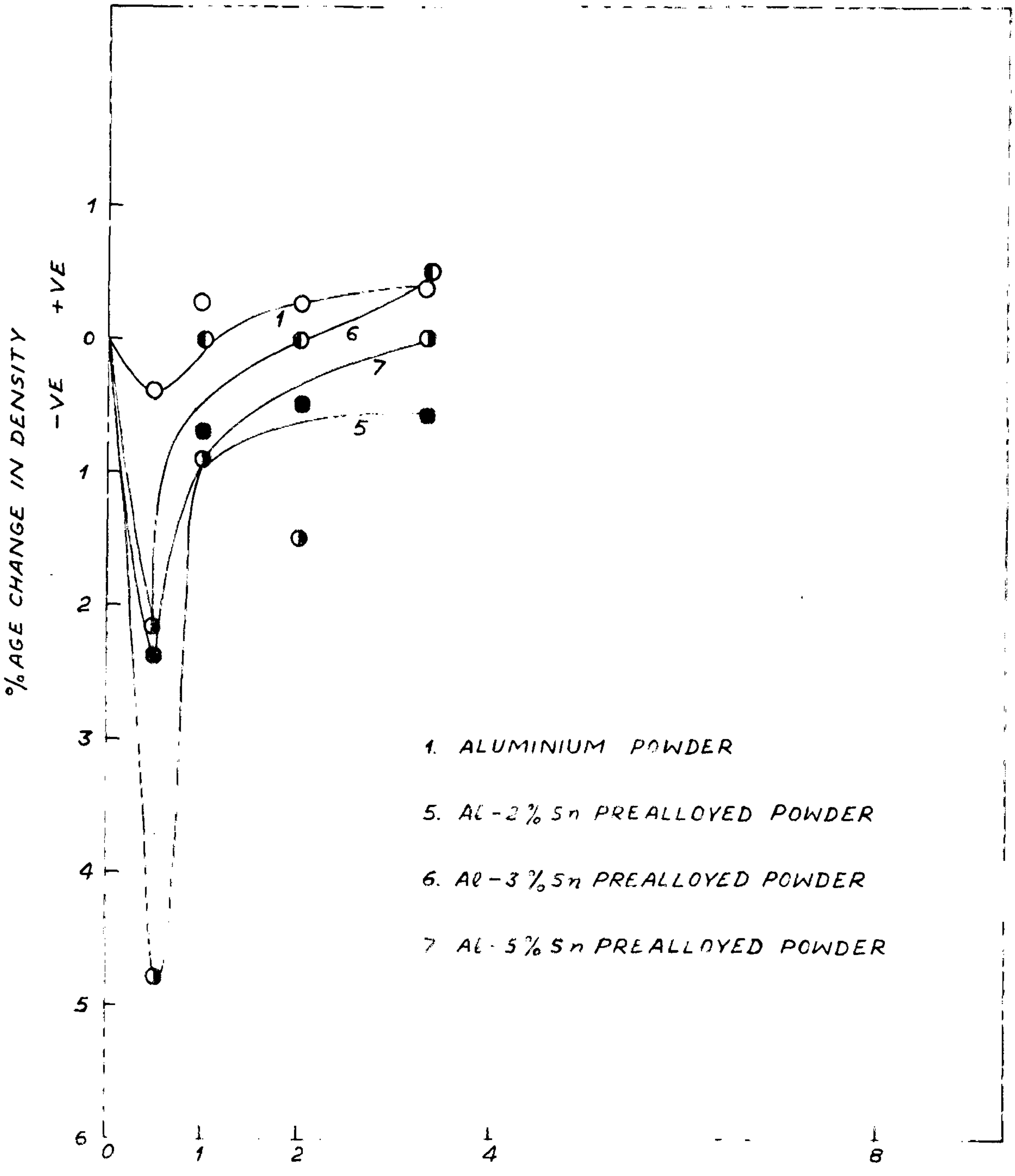


FIG.29 DENSITY CHANGE OF ALUMINIUM AND ITS ALLOY POWDER DURING SINTERING AT 600 °C

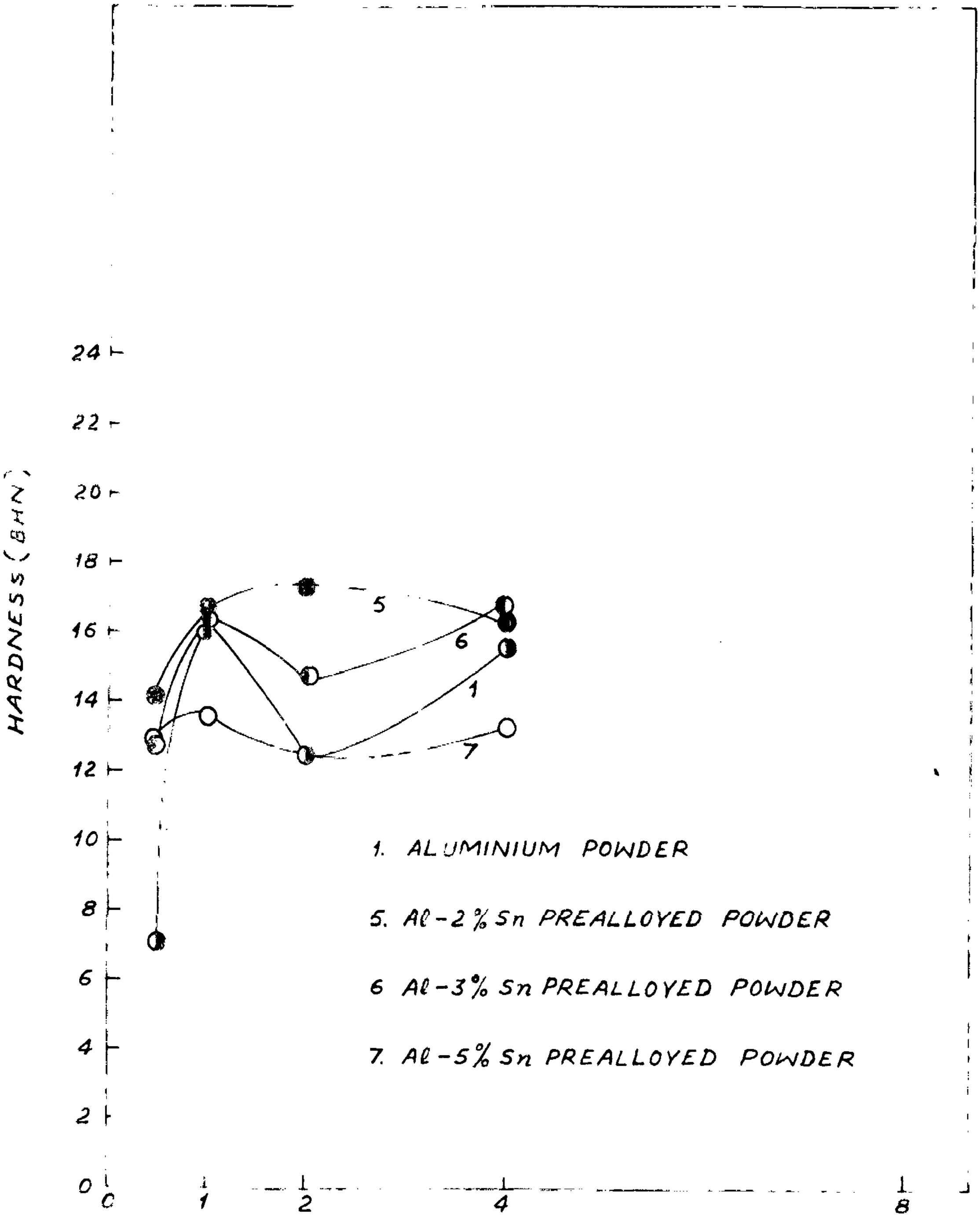


FIG.30 HARDNESS OF ALUMINIUM AND ITS ALLOY POWDER DURING SINTERING AT 600°C

In the present investigation atomization of molten aluminium , aluminium tin and aluminium copper alloys have been undertaken. As reported elsewhere the atomizing medium was cold air blown through a suitably designed nozzle. After the powder of different compositions produced, they were fractionated with the help of set of sieves and the results of such screen anolyses have been plotted in Figure (22, 23, 24) and table (1, 2 and 3). A suitable size distribution mentioned in table 4 was taken and the powder mass was compacted to a suitable pressure, which is normally required for producing porous metal bearings containing 20-25% porosity. The compacts produced this way were sintered at 600°C and the optimum sintering conditions were established through expansion and hardness data. For the comparison of sinterability of the atomized powder and for the purpose of improving this sinterability, two separate sets of observations were made. Firstly, individual metal powders were mixed resulting in the same compositions as that of atomized alloy powders and properties of sintered compacts produced out of these powder mixtures were studied. Secondly, atomized alloy powders were subjected to grinding operation just before compaction employing alcohol as a grinding medium to avoid any chance of possible oxidation of freshly created surfaces. Sinterability of such ~~powder~~ powders as reported in the subsequent pages is found to be better than ordinary atomized alloy powders. It is also clear from the results mentioned below that atomized alloy powders have better sinterability than the mixture of metal powders of the same compositions. Also a ternary addition of copper in aluminium-tin alloy leads to better sinterability.

Size Analysis

Size analysis results of Al-Sn prealloyed powder are plotted in fig. 22 as cumulative percentage oversize vs sieve opening. Curves show that on increasing percentage of tin, percentage of finer size goes on increasing i.e. curves shift downward parallel to that of aluminium.

Spacing between the curves representing pure aluminium and Al-2% Sn alloy is more than the spacing corresponding to Al-3% Sn or Al 5% Sn alloys. Similar results are also obtained for Al-Cu alloy compositions as shown in Fig. 23. However, the effect is not that much marked as it was seen in the case of Al-Sn alloy compositions.

Fig. 24 gives even better idea of an yield of particular powder size when plotted against the % alloying element. It is clear from the curves that for both, aluminium-tin and aluminium-copper alloys, the yield of -100 mesh powder increases initially and curves become horizontal later on. Also slope of the curve for aluminium tin compositions is more than that of aluminium copper compositions.

The effect of alloying elements on the yield of particular particle size is mainly determined by the modifications in viscosity and surface energy values of an alloy. Former relates to the amount of flow of the molten metal or alloy while the latter one concerns the amount of kinetic energy required to ~~distinguish~~ disintegrate the molten metal stream in the form of the fine droplets and thereafter particles. Thus increased viscosity leads to lower flow rate of the molten metal or alloy and hence finer plarticles. Similarly large value of surface energy acts as a barrier for creating new surfaces and hence relatively coarser particles are resulted.

By the addition of tin or copper in molten aluminium, viscosity in the former case decreases slightly while in the latter case increases sharply this is clear from liquid and solid solubility data ofSn and Cu in Al and also from the fact that binding force between Al and Cu atoms is much more whereas between Al and Sn atoms it is practically negligible. Thus from the point of view of viscosity, Al Cu alloy should lead to a finer particle size than Al-Sn alloy. On the other hand, the surface energy values for Al-Cu compositions higher than Aluminium which leads to coarsdr powder. In case of Al-Sn, the surface energy value is lower than aluminium which is favourable for finer size of particle. Results show that addition of tin or copper to aluminium reduces the particle size of powder. It indicates that effect of increase viscosity for Al-Cu composition and effect of decrease surface tension for Al-Sn composition is pronounced.

Shape Study

Aluminium and its alloy powder is studied under stereomicroscope at a magnification of 40 X. Aluminium powder appears elongated in one direction with pointed end and bended from the middle. Their surface appears to be rough. Al-Cu prealloyed powder appears more elongated and pointed as compared to that of aluminium converse is observed in case of Al-Sn prealloyed powder, surface of Al-Gu prealloyed powder is seen more smooth as compared to that of aluminium or Al-Sn prealloyed powder.

Different size fractions of sieved powder were studied and itwas found that there was no change in shape with respect to difference in size of the powder.

Al-Cu prealloyed powder are seen more elongated in one direction and overall smooth surface throughout. This is due to large overheating (liquidms temperature of Al-4% Cu is lower than that of aluminium) which results to drop to solidify after sufficient time has elapsed, and surface tension force will act for larger time to smoothen the particle surface. Also large surface energy of Al-Cu alloy tends to minimise the surface resulting into smooth spherical surface of the particle, but drop has been elongated in one direction due to difficulty of flow caused by large viscosity of alloy. Less elongation and rough surface of particle of Al-Sn powder are due to its low surface tension and viscosity.

Sinterability

While studying the sinterability of two alloy system namely Al-Sn and Al-Cu it is obvious that for tin compositions ~~that~~ tin phase liquifies during sintering whereas for Cu composition boing within the single phase region, it remains purely a solid state sintering. Thus the mechanism of sintering would be different in these alloy system.

In order to study the sinterability of these systems two variables have been taken here namely changes in density and change in hardness with respect to the sintering time keeping temperature constant to a level of 600°C. The sintering atmosphere was vacuum of the order of 5-7 microns as reported earlier.

Density Changes

Change in density Vs. sintering time as shown in fig. 25, 26, and 27) indicates that for Al-Sn composition there is an initial decrease in density. This decrease is even more in the case Al-Sn atomised alloy powder in comparison to Al-Sn mixed powder. However, no such decrease was observed in case of Al-Cu system.

The decrease in the density of Al-Sn composition may be due to an hinderance in the process of degasification because liquid tin phase might obstruct the capillary channel of inter connected porosity and thus causing the gases contained in the pore to expand.

After such an initial decrease there is continuous increase of density in all the cases. The reason of which appears to be the decrease a number and sizes of pores. Higher densification is observed in the case of Al and Al-Cu systems as compared to that of Al-Sn. This is obvious because the binding forces between Al and Cu and also between Al-Al atom are more compared to that between Al-Sn atoms.

While studying the effect of Cu on density changes it is clear from fig. 27 that addition of Cu has resulted to lower density values. Thus from the point of density changes addition of Cu has not been beneficial in improving the sinterability. Effect of grinding just before the compacting was carried out in the case of pure Al and the results shown in fig. 25 indicate that it has resulted in better densification after sintering.

Hardness Changes

The general characteristic of hardness curve shown in fig. (28, 29 and 30) shows that hardness increases from 1/2 to 1 hr of sintering in the all cases. However, after such an initial increase there occurs a minima in all the cases. In the case of Al-2% Cu prealloyed powder the initial increase of hardness remains continuous and it is only after 8 hrs. of sintering that a slight decrease in hardness is observed.

The mixture of Al-Sn powder shows a very poor hardness, However, the nature of curve remain the same as that of Al-Sn prealloyed powder. Furthermore addition of copper seems to improve hardness value when compared with binary alloy, in the final state of sintering.

Tho first increase in hardness curve referred above is due to increase in number of bonds and densification through elimination of pores. In this respect both hardness and density changes are alike. The occurrance of minima in the curve indicate the possible grain growth of aluminum matrix as a result of which hardness decreases and the density increases. However, this grain growth lattor on envelops the porosity leading to an increase in further density and thereafter again there is general increase in hardness as shown in the curves.

In Al-Sn prealloyed powder, higher hardness value is observed at 2% tin. This may be due to fine distribution of tin in aluminium matrix which results dispersion hardenging.

At more than 2% tin, lower hardness value may bedue to presence of large softer tin phase.

The poor hardness values observed in the case of unalloyed powder mixtures may be due to nonuniform distribution of tin in case of Al-Sn composition and in complete formation of Al-Cu alloy in case of Al-Cu composition.

CHAPTER - 6

CONCLUSION

The following conclusion may be drawn from the preceding chapter.

(1) Addition of tin or copper to aluminium helps in the production of finer powder than is possible with Aluminium alone. In this regard tin is more effective than copper.

(2) Grinding of powder mixture in presence of methyl alcohol prior to compacting helps in obtaining higher densities of sintered compact.

(3) After sintering, Aluminium-tin and Aluminium-copper pre-alloyed powders have higher densification and hardness values as compared to Aluminium or Aluminium-tin or Aluminium-copper powder mixtures.

(4) Addition of copper to aluminium-tin powder mixture improves the hardness after sintering.

CHAPTER - 7

SUGGESTIONS FOR FURTHER WORK

The work can be extended for economical production of atomised powder of better quality. By quality of powder it is meant that powder should be free from undesirable impurities such as oxygen and it should possess properties desirable for compaction and sintering. Efficiency of atomization is measured by fineness of powder particle. Along the following lines work can be extended.

1. Oxidation of powder takes place in atomising zone when air and powder in hot condition are in contact. Therefore, for prevention of oxidation it is required to consider the atomizing media. Two types of atomizing media are possible, (liquid and gas) for atomization. In case of liquid as atomizing media, the process may not be efficient where lot of heat loses from drop occurs by atomizing media in its heating and vaporisation in atomizing zone. Gaseous type atomizing media have been proved to show economy in atomization process. For prevention of oxidation, instead of air other gases such as petrol gas,

steam and nitrogen inert gases may be tried.

(2) Close circuit atomization is also possible to reduce the chances of oxidation where the atomizing media is circulated reppatedly using vacuum and compression devices in between and media is kept free of O_2 content throughout the process.

(3) For obtaining better efficiency and contool on atomisation process variables such as rate of metal flow and air flow, pressure of air and temperature of metals require a critical study.

Apart from working in direction of economically production of better quality powder, some other possible aspect of atomization may be studied which are as follows :-

(1) Use of mixture of petrol vapour and air in particular proportion may be considered as atomizing media.

(2) Use of multi-cone cavity nozzle which will supply gas at different angles with different velocity, may be useful for obtaining powder of particular size distribution economically.

(3) Use of metal wire instead of using molten metal and atomization of it by ignited mixture of air and petrol in proper proportion may have a good approach for atomization process. However, economy is yet to be tested.

There are also some suggestions for further work to improve the sinterability of aluminium and aluminium base alloy powders which are as follows :

(1) Addition of activating agents like fluorides of calcium sodium and lead etc.

(2) Sintering under ultrasonic vibration to remove oxide at the particle surface.

(3) Sintering by fluctuating temperature very close to melting point of aluminium or liquidus of aluminium base alloys.

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