Production of Tin Powder Using Gas Atomization Process

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Abstract:
Gas atomization process is widely used for the production of various metal and pre-alloyed powders. The process involves the disintegration of a liquid stream of molten metal into liquid metal droplets by the impingement of high pressure gas jets. The liquid metal droplets subsequently cool and solidify into metal powder particles, which can typically range from 1 to 150 µm.
After a detailed study of various atomizing processes, the cross jet gas atomizing scheme was selected for the present research work. Experiments were carried out using air and argon gas as atomizing media. Particle shape was observed using Scanning Electron Microscope. The particle size distribution was measured using the Laser Particle Size Analyzer. XRD analysis of different powder samples was carried out to measure the oxygen contents. Spherical shape particles ranging from 1 µm to 60 µm sizes were produced.
Key Words: Gas atomization, cross jet gas atomizing, atomizing media, laser particle size analyzer, XRD analysis.

1. Introduction:

Various metal powder production techniques are used to manufacture a large variety of metal powders to meet the requirements of market all over the world. It is estimated that worldwide capacity of atomized powder has been increased by 10^6 metric tons per year. Atomization is the most economical method for production of metals and pre-alloyed powders [1]. The gas atomization process remains a good choice among the different methods of metal powder production due to its versatility, the quality and purity of the obtained powder and the potential for mass production. One of the main advantages of the gas atomization processes is that it can produce high quality pure metal and pre-alloyed powders as well [2]. In the gas atomization process, high pressure jets of gas (air, argon, nitrogen or helium) is used to disintegrate a stream of molten metal into small droplets which further solidify in fraction of seconds by the quenching affect of gases. Different types of nozzles can be designed to maximize the production rate and to control the characteristics of powders.

Depending on a particular design, there are generally two types of gas atomization configurations that are used in low and moderate pressure powder metallurgy production. These are known as:

1- Open stream or free fall and cross jet atomization
2- Closed, confined feed or close couple atomization

After a careful observation and consultations, cross jet gas atomization technique has been selected for this research. It is easy to use and manage this design during atomization. In the cross jet atomization, the molten metal and gas stream are at right angle to each other. The suction is provided by the high pressure stream jet at the end/tip of atomizing nozzle [3]. The schematic diagram of this design is given in Figure 1.

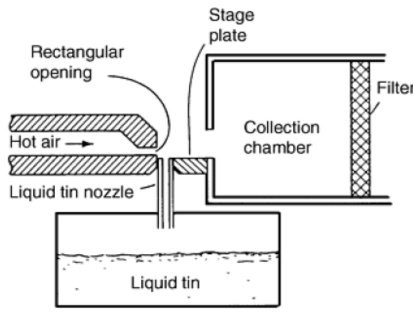

Figure 1: Cross – jet gas atomization scheme. [3]

Due to lake of technical knowledge and requisite facilities, metal powders are not being produced in Pakistan. These powders are imported to fulfill the local industry requirements. Thus, there is a need to explore the field of metal powder production to meet the local demands. The main objective of this study was to develop a laboratory scale experimental set up for the production of low melting metal (such as tin) powders through gas atomization.
Tin have many industrial applications including production of porous self lubricating bronze bearings, brazing pastes, as constituents in solders and its powder. The powder metallurgical applications include brake linings, metal graphite brushes, structural parts, clutches, friction disks, bronze filters, abrasive grinding wheels, and chemical formulations, additives for plastics and rubber and to make tin flakes [3, 4].

2. Experimental:

A cross jet gas atomizing scheme was selected for this study. Experiments were carried out using air and argon gas as atomizing media. Electric heating furnace was used for melting tin metal with superheat of ~ 50 ^{o}C. A small chamber having dimensions of 8 ft length, 4 ft height and 3 ft width was fabricated for collection of metal powders. Particle shape was observed using Scanning Electron Microscopes. The particle size distribution was measured using the Laser Particle Size Analyzer. XRD analysis of different powder samples was carried out to measure the oxygen contents.

3. Results and Discussion:

3.1 Selection of Atomizing Design:

For tin powder production, the horizontally cross-jet gas atomization technique is often used [1]. Keeping in view the complexity of all available options of nozzle designs, cross jet gas atomizing scheme was selected for this research study. The main reasons of its selection include easy handling, economic factors and its application for low melting metal powder production.
In this study, the following arrangements were made for designing of the atomizing set- up.

3.1.1 Nozzles:

Some sort of arrangement is required to supply molten metal and high pressure gas to accomplish atomization. Nozzles are required to be designed and installed in the apparatus. The nozzles used in the commercial atomization are made up of ceramics to withstand high temperature and resistance to wear under the impact of high pressure jets of fluids. In this study, the metal has a low melting point that’s why stainless steel pipes of 4 mm internal diameter have been employed to carry molten tin metal for atomization. Same material has been used for the supply of atomizing gas from a polyurethane pipe to atomizing ends. As stainless steel has much high melting temperature as compared to tin thus, it can be used for a short period and experimental purpose.

After performing experiments on water, the angle between molten metal and gas pipes was adjusted for appropriate and efficient atomizing. As both pipes are at right angle to each other which infect is the requirement of this design, some sort of changing in the angle could be done as it has been kept flexible in the design, Figure 2.

Figure 2: Nozzles design used during experimentations.

3.1.2 Prevention of Metal Solidification in Nozzles:

During the initial experimentation, it was observed that the solidification of metal in stainless steel pipes created many problems. To avoid such solidification, pre-heating of the pipes was carried out using heating coils all around these pipes. The heating through coils again was unsuccessful because due to concentration of heat in the middle caused steel tubes to be melted. However, pre-heating of the pipe with a gas flame provided good results.

3.1.3 Atomizing Gas:

During initial experimentations, air was used for atomization. This produced powder having dull color, which is a clear indication of oxidation. But later on to avoid oxidation, a chamber was fabricated and inert gas (argon) was used for the atomization. The pressure of gas kept constant at 20 bars.

3.1.4 Melting Facility:

Electric heating furnace was used for melting. The melting temperature of tin is 232 ^{o}C and super heating of 50 – 60 ^{o}C was given to compensate any heat losses during transfer from furnace to atomizing stand. The viscosity of melt and super heat temperature plays an important role in controlling the particle size distribution of powder produced.

3.1.5 Atomizing Chamber:

Powder collection chamber is a main part of an atomizing unit. A small chamber having dimensions of 8 ft length, 4 ft height and 3 ft width was fabricated for collection of metal powder. It was then observed that length is much more important in order for the tiny powder particles to fall freely rather than striking at the opposite end and get deformed. Transparent acrylic plastic sheets were used for the construction of atomizing chamber. The main supports and structure was provided by the mild steel strips. Some openings were left at the end to produce a positive draught for the exit of atomizing gas. This will not allow the air to come inside the chamber as pressure inside is more than outside during experiments. In order, to make easy collection and separation of different particle sizes, eight partitions were incorporated in the bottom of this powder collecting chamber. They were designed in such a way, that powder particles can slide along the walls of these partitions and ready to collect at the bottom from drawable semi- cut PVC pipes. Powder can be collected from this semi-cut pipes after withdrawal of them from chamber as shown in Figure 3. After removal of the powder, they can be reloaded into the chamber assembly. As one end this chamber is open to environment and there is a strong potential of air to come inside and oxidize the metal particles during atomization. This can be minimized by flushing the chamber with argon gas before starting the atomization.

Figure 3: A view of the atomizing chamber and tin metal powder in one partition of collection slot in the chamber.

3.2 Powder characterizations:

Following steps were taken for the characterization of the tin powder produced:

3.2.1 Sampling:

It is the most important step during any physical and chemical characterization of any powder. In order to avoid any misleading results, a true representative sample of powder should be taken according to the MPIF Standard 1.

3.2.2 Particle shape:

Particle shape was observed using Scanning Electron Microscope. Two of the representative micrographs are given below which show that most of the particles are spherical, Figure 4.

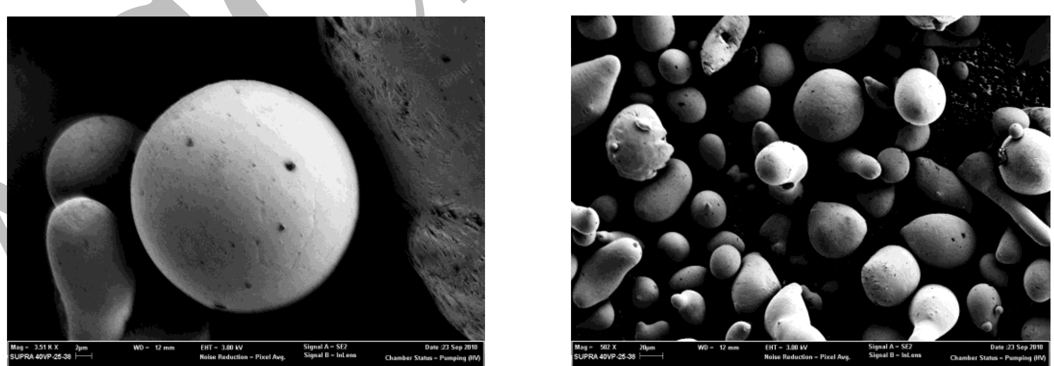

Figure 4: Scanning electron micrographs of gas atomized tin powder.

3.2.3 Particle size distribution:

Particle size distribution of powder determines the behavior of particles during processing and later on the mechanical properties of sintered product. Traditionally, sieving and screening is the method used to determine the particle size distribution of any kind of metal powder in powder metallurgy. It can be efficient when there is a large amount of powder less than – 80 mesh i.e. smaller than 180 µm and only a small quantity is less than 10 µm. In the application where large quantity of metal powder fall below 38 µm, these sieving and screening become less reliable and efficient, then other more modern and sophisticated methods are employed. The electronic methods developed have high accuracy, measurement range and less time consumption and hence less labor work. But again there is always a discrepancy in the exact size in diameter measurement by all methods available. The particle size distribution was also measured by using the laser particle analyzer which works on the principle of Fraunhofer diffraction theory. The results of one of the test are given in Table 1.

Table 1: Particle size distribution of tin powder produced.

Particle Size, µm	<58.5	<35.6	<21.4	<10.5	<5.6
Fraction, %	93.5	71.3	52.8	15.6	6.8

3.2.4 Chemical Analysis:

Chemical testing is required to check the purity and the amount of oxidation which takes place during atomization. The samples of powders atomized in argon gas were subjected to this test. One of the results of XRD analysis is shown in Figure 5, which shows the presence of some oxygen contents. Thus, to avoid the oxidation the atomization should be carried out in a closed chamber under a neutral atmosphere. Some amount of aluminum is also observed. Impurities in the tin melt stock carry over to the powder. Thus, the tin powder purity is directly related to the purity of the ingot melted [5].

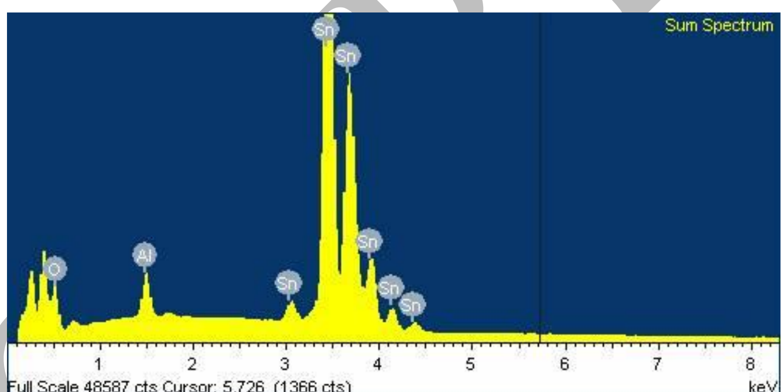

Figure 5: XRD analysis of gas atomized tin powder.

4. Conclusions:

- The designed and fabricated pilot scale gas atomizer was proved effective for producing tin powder.
- The result of the present study illustrates that low melting metal powders such as tin may be produced using cross jet atomization technique.
- As stainless steel has much high melting temperature as compared to tin thus, its pipes can be used for supplying molten metal and gas jets.
- Pre-heating of the pipe with a gas flame was found useful for avoiding solidification of molten metal in the pipe line.
- The size of the atomizing chamber should be large enough to allow powder particles to fall freely rather than striking at the walls of the chamber and get deformed.
- The powder particles showed typical spherical shape of gas-atomized powders.

- To avoid the oxidation of the powder particles, the atomization should be carried out in a closed chamber under a neutral atmosphere.

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