Receiving finely divided metal powder by inert gas atomization

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Abstract: In this study, the inert gas atomization method to obtain finely divided metal powder was studied. Two different configurations of inert gas atomizers were compared to identify the most appropriate one to obtain high spherical AM powder. Some problems of free-fall atomizer were discussed.

The method of obtaining small and medium-sized spherical shape metal powders using dispersing of melts called atomization [1]. This process occurs in special installations called atomizers. Spraying (atomization) is widely used in the production of powders of multicomponent alloys, with an amorphous structure that allows to achieve a uniform chemical composition, even with a content of doping components above their solubility limit in the main component of the alloy. In addition, the powders obtained using the methods for dispersing melts have a particle shape that is close to spherical. More than 90% of all powders used in additive technologies are obtained by melt dispersion methods. One of the main methods is gas atomization [2].

The main reasons for using this spraying technology are:

- The possibility of high bandwidth and the disintegration of high mass costs
- Greater amount of heat exchange between the gas and the particles for rapid partial cooling of the particles
- Direct delivery of kinetic energy to accelerate particles towards the substrate / deposition for compaction
- Minimizing the risks of oxidizing sputtered materials during spraying using inert gases.

According to this technology, the metal is melted in the melting chamber (usually in a vacuum or inert medium) and then drained in controlled mode through a special device - a sprayer, where the liquid metal flow is destroyed by an inert gas jet under pressure. Spraying can occur in water or in an inert gas stream. Water has the advantage of rapid heat transfer, but there is a high risk of hydrogen and oxygen generation during spraying [3]. During the flight phase, the particles undergo a spherical shape under the action of surface tension forces. To obtain fine powders d = 10-40 microns, most often used in additive technologies, the VIM-atomizers (Vacuum Induction Melting) are used in which the melting chamber is evacuated to minimize melt contact with oxygen and nitrogen. And the technology itself for obtaining powders using vacuum melting machines is called VIGA-Vacuum Induction Melt Inert Gas
Atomization, that is, the "technology of gas atomization of a metal melted in a vacuum chamber by induction heating".

VIGA atomizers are used to produce the following powders:

- Nickel refractory alloys (Inconel 718, Rene 88, etc.) for parts of aircraft and stationary turbines;
- Alloys based on cobalt for use in medicine, dentistry and production of ion sputtering targets;
- Powders for plasma deposition (NiCrAlY, CoCrAlY, etc.) for plasma spraying of protective coatings on parts made of heat-resistant alloys;
- Powders for granule metallurgy (17-4 PH, 316L) for automotive parts of mass production;
- Sintering compositions in the powder layer (eg cobalt alloys, precious metals) for use in AM machines;
- High-alloy steels (tool steel, quick-cutters) with very high carbide content;
- Non-ferrous metals (copper or tin alloys for various applications).

The design of the classic free fall atomizer is simple and very reliable. Moreover, such sprayers have several applications. They are used, for example, in spray dryers, in powder manufacturing devices or in spray forming processes [4]. Figure 1 shows a sketch of a conventional external atomizer of free mixing. The design is based on the combination of two systems of gas nozzles, called the primary and secondary gas nozzles [5]. Secondary nozzle is the main spraying unit. Concentric jets of gas from the secondary nozzle fall on the central jet of the liquid, which decays due to instabilities from the shear action of the secondary gas stream and its relative velocity [6]. The process of disintegration of the liquid jet in the spraying zone is under the secondary gas nozzle [7]. Due to the splitting of the alloy jet into droplets already inside the spray gun body, this design is mainly used for spraying viscous or high-temperature liquids and melts.

![Figure 1. Main areas of the free fall atomizer](image-url)

In this method, both rotary crucibles and crucibles with bottom draining are used. For melting, ceramic or graphite crucibles can be used. Depending on the material of the crucible, the melting point can reach 1900 °C. The molten metal is poured into a special receiver, to which an inert gas is supplied.
under pressure (usually argon, sometimes nitrogen). The process of metal spraying has three phases - initial, working and final. In the initial phase, the system enters the operating mode: the metal drain valve is opened (it takes some time to stabilize the flow), the supply of the spray gas is switched on, in a precisely defined ratio between the amount of metal and gas. This phase lasts a few seconds.

A common characteristic of the two spray nozzle variants used for sputtering molten metal is the vertical outlet of the melt jet from the casting device through (in most cases cylindrical) molten nozzle in the direction of gravity. Also, in most cases, the central jet stream of the melt is surrounded by a gas flow from one (slotted) configuration of the jet or a set of discrete gas jets that flow parallel to the direction of melt flow or within the angle of inclination of the attack to the melt stream. A coaxial gas atomizer usually leaves the atomizer at high pressures with high kinetic energy.

In the process of spraying molten metal, two basic configurations and types of two-fluid nozzles must be distinguished. The first type is called a closed or closely-coupled atomizer, and the second type is called a free fall atomizer. Both concepts are illustrated in Figure 2.

![Figure 2. Principles of atomization of a gas of melts: atomizer with a closed connection (a) and a free fall atomizer (b)](image)

Then the working phase begins, in which the metal drainage process is stabilized and the required ratio of metal and argon consumption in the atomizer is achieved. At the end of the working process, the rate of metal exit from the crucible decreases, the flow parameters change, and the balance between the mass ratio of the metal and the gas is disturbed. This process also lasts a few seconds. In the first and final phases, the powder turns out to be substandard. Therefore, to increase the productivity and efficiency of the system, it is necessary to increase the proportion of the working phase in the overall balance of the atomization cycle time.

The atomization unit, where the process of metal sputtering actually takes place, is a very complex device that, for structural and technological reasons, can not be performed with dimensions that are less than certain. For qualitative spraying, a certain ratio between the metal consumption and the gas consumption must be maintained [8].

Also, due to the slope of the secondary gas stream, a recirculation gas flow region may occur under the secondary gas nozzle, depending on the design of the atomizer and the operating conditions. If the intensity of the recirculation flow is large enough, liquid bundles or droplets are transported back to the spray gun body. Here, these adhering fragments can clog the gas and / or liquid openings and adversely affect or even stop the spraying process. In conventional free-fall atomizer designs, the recycle gas stream is suppressed by the primary gas nozzle. The primary flow of gas merges with the liquid stream, thereby directing the liquid into the spray zone without recirculation.
The flow of gas in a atomizer with close coupling immediately closes the outlet of the melt. Within the confined atomizer, the distance between the gas outlet and the melt flow is much less than in the free fall device, where the melt stream moves a certain distance in the direction of gravity before the gas flow falls onto the central jet of the melt. A close-coupled configuration typically tends to give a higher spray efficiency (in terms of smaller particles with the same energy consumption) because of the lower distance between the gas and melt yields. But a closed type of atomizer is more susceptible to problems of melt freezing on the tip of the nozzle. This effect is due to the wide cooling of the melt by the expanding gas flow, which leaves in a close connection near the melt flow. During the expansion of the isentropic gas, the temperature of the atomization gas decreases (sometimes well below 0 ° C). Because of the close spatial relationship between the gas and melt fields, this facilitates the rapid cooling of the melt at the tip of the molten nozzle.

The problem of freezing is relevant, in particular, for spraying. In this process, a discontinuous periodic operation is performed in all technical applications (for example, due to the periodic preparation of the melt or the limited preform that extends to the formation of the spray). The operating time of spray forming processes ranges from a few minutes to about one hour. The problem of the thermal dependence of freezing is most important in the initial phase of the process itself, when the melt flow leaves the nozzle for the first time. At this point, the nozzle tip is still cold and must first be heated, for example by a hot melt flow. This heating process lasts a certain time. Therefore, in the first few seconds of the melt spraying process, there are often problems with freezing associated with heat treatment.

In addition, there are often problems associated with chemical or metallurgical processes in melt delivery systems. Not all these problems have been solved at the moment. A number of problems arise due to a possible change in the composition of the melt material or material of the intermediate ladle or nozzle, due to possible melt / spill response or melt segregation effects from diffusion. This kinetics of the process is somewhat slower than the kinetics of the thermal freezing process mentioned above and can contribute to operational problems in the next stages of the process. [2]

Free fall atomizers are much less problematic than atomizers with close coupling, because the flow of melt streams and the gas stream are well separated at the outlet from the tundish. Therefore, the cooling of the melt with cold gas occurs later than in atomizers with a close bond. As an additional advantage, there is the possibility of controlled mechanical or pneumatic scanning and, consequently, oscillate the gas atomizer with respect to one axis. Also, for the spraying, the concepts of spatial / temporal distribution of the gas jet segments within the nozzle can be used. Thus, the free fall atomizer gives the operator an additional degree of freedom in controlling and controlling the distribution of the mass flow of droplets in the atomizer. This is the most important physical property, since in other systems the nozzle can only affect the pressure of the atomizer gas (during the working process). With controlled scanning of the nozzle, the mass flow can also be distributed over a certain area (necessary, for example, to form flat products). [10]

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