INVESTIGATION OF THE FABRICATION PROCESS OF METAL 3D PRINTED POWDER BY MOLTEN ALLOY DISPERSION METHOD USING CENTRIFUGATION AND HIGH-PRESSURE GAS

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DOI: 10.17973/MMSJ.2023_10_2023059

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Atomization using centrifugation and high-pressure gas is considered to be a relatively new method of producing metal 3D printing powders. The metal powder made by this method is expected to obtain an adequate small-scaled size with the limitation of satellites stuck to the surface due to the reduction of the gas flow in the dispersion chamber. This work conducted experimental studies with aluminum alloy graded of AlSi10Mg. The particle size distribution of the fabricated metal powder after fabrication was evaluated using standard sieves and the laser diffraction method. In addition, the morphological structure of metal powder particles was examined by scanning electron microscopy method. The investigation results have determined the influence of geometrical and technological parameters of the atomization process, including liquid metal flow, liquid metal overheating, and some structural parameters of equipment on the quantity and quality of the product.

KEYWORDS

3D printing, High-pressure gas, Metal powder, Atomization

1 INTRODUCTION

Nowadays, for metal 3D printing technology, the quality of input material is supposed to be a strict requirement. Currently, one of the main obstacles to the widespread implementation of this technology in production is the high cost of metal powders – the input materials for 3D printing because they must meet tough technical criteria: 1. Highly concentrated particle size with particle size from 10 to 150 µm; 2. Spherical grain profile; 3. Chemical composition without or with few impurities [1, 2]. The solution to this problem is none other than mastering the technology of metal 3D printing powder manufacturing. According to the publicized works [2, 3, 4], over 90% of metal powders for 3D printing purposes are made by liquid alloy dispersion method with dispersing agent, usually inert gas (gas atomization), centrifugation (centrifugal atomization), or variations of the two methods above. This study uses a metal powder fabrication method using a combination of centrifugation and high-pressure gas. This is considered to be a relatively new method of making metal powders. Still, it has proven the high duality, allowing powders of materials of different characteristics, including those with high viscosity, low surface tension, or low density, such as aluminum alloys [3, 4]. The working principle of the method is shown in Figure 1.

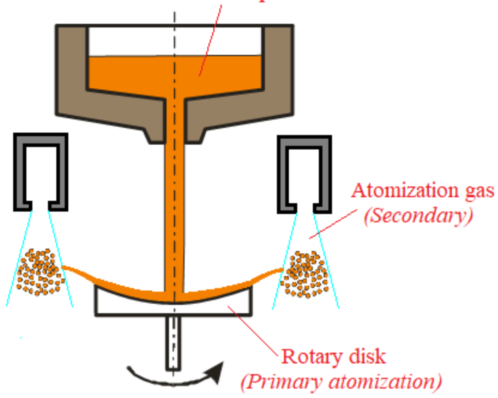


Figure 1. Diagram of dispersion of molten alloy using centrifugation and high-pressure inert gas

The essence of this method can be clarified as follows. A centrifugal rotating disc is used to create primary dispersions for the liquid metal to increase its specific surface energy; as a result, the liquid metal will take the form of a thin cone-shaped film. According to inertia, the liquid metal at the end of the primary dispersion phase will move to the working area of the dispersed gas and continue to be dispersed by the gas streams in a state of momentum and high pressure. In this second dispersion stage, the liquid metal is atomized to turn into smaller spherical droplets.

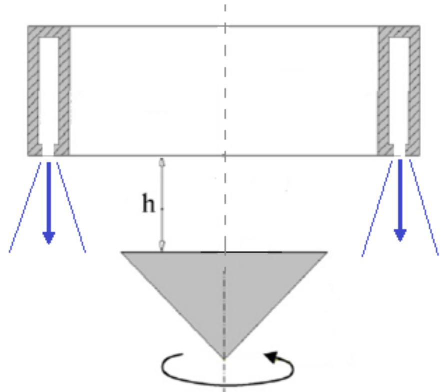


Figure 2. Illustration of geometrical parameters of metal powder fab equipment

Although molten alloy dispersion technology has been used in metal powder production since the 60s of the last century, studying the movement and dispersion of metal streams at high temperatures is still a challenge today. The urgency of the problem is evidenced by many patents, doctoral theses, and scientific works related to metal powder dispersion devices and related processes [1, 2, 10, 11]. On the other hand, the relationship between the geometrical parameters of the device and the technological parameters of the dispersion, and the properties of the metal powder formed has not been fully studied. In this research, we have conducted experimental work of metal powder fabrication at different conditions by varying technical parameters such as dispersion temperature, liquid metal flow, and distance from the top surface of the centrifugal disc to the outlet of the gas nozzle. Therefore, we are able to investigate the influence of those parameters on the quality of the fabricated metal powder.

2 EXPERIMENTAL WORK

2.1 *Input materials and chemicals*

In this research, the input material is aluminum alloy grade AlSi10Mg provided by the Vietnam National Institute of Mining - Metallurgy Science and Technology /Ministry of Industry and Trade of Vietnam. The chemical composition of aluminum alloy AlSi10Mg is presented in Table 1.

Table 1. Chemical composition of aluminum alloy AlSi10Mg

	Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn
Ave %	9.9811	0.5273	0.0276	0.3495	0.3772	0.0473	0.0483	0.0988
	Ti	Bi	Co	Ga	Sn	Pb	Be	Al
Ave %	0.1202	0.0378	0.0415	0.0297	0.0449	0.0870	0.0500	87.8774

The dispersion gas used in the research is Argon gas, with a purity of 99.999%, in the form of a 40-liter compressed air cylinder at a pressure of 150 bar.

2.2 *Fabrication equipment*

The liquid alloy dispersion process is carried out on a metal 3D printing powder fabrication which is constructed by the research team. (Figure 3). The technical parameters of the equipment are presented in Table 2.

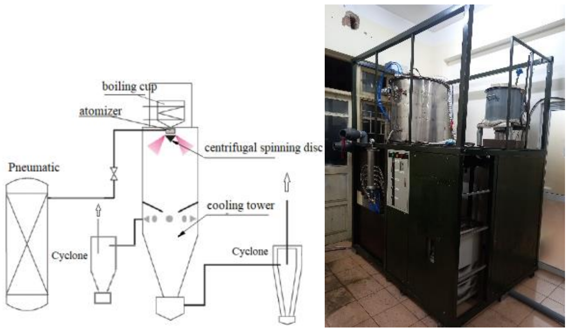


Figure 3. Technological diagram (left) and actual image (right) of metal 3D printing powder fabrication equipment.

Table 2. Technical parameters of metal 3D printing powder fabrication equipment

Technical parameter	Value
Kiln volume,	2 litters
Maximum vacuum pressure	-600 mm Hg
Working temperature of the kiln	300 ÷ 1100 °C
Dispersion gas pressure	≥ 24 atm
Dispersion gas flow	~ 0,96 m^3/min
Centrifugal disc speed,	15000 rpm

2.3 *Experimental parameters*

In this study, the experimental parameters of the metal powder fabrication process were investigated, including:

- *Dispersion temperature.* The aluminum alloy AlSi10Mg has a melting point of 590 °C. According to the published data in other works [6], the liquid alloy dispersion temperature is about 100 to 200 °C higher than the melting temperature, depending on the nature of the investigated metal and the dispersion equipment. Therefore, in this study, the research team investigated the liquid alloy dispersion process at the temperature range from 700 to 900 °C.

- *Liquid metal flow*. This parameter is adjusted by changing the inner diameter *d* of the liquid metal conduit with various values of 3mm, 4mm, 5mm, 6mm, and 7mm, respectively;

- *Distance from the top surface of the centrifugal disc to the outlet of the gas nozzle* (**Figure 2**). The proper distance *h* would determine the area where the kinetic energy of the dispersion gas is at the highest peak. It is also the destination where the liquid metal, after the primary stage of dispersion using centrifugation, would arrive by inertia and be dispersed by the dispersion gas into smoother droplets. According to our survey results by simulation [9], the distance from 1 to 1.5 cm would give the best dispersion effect. In this study, the experimental surveys will be conducted with various values of the distance *h*: 0.5, 1, 1.5, 2, and 3 cm.

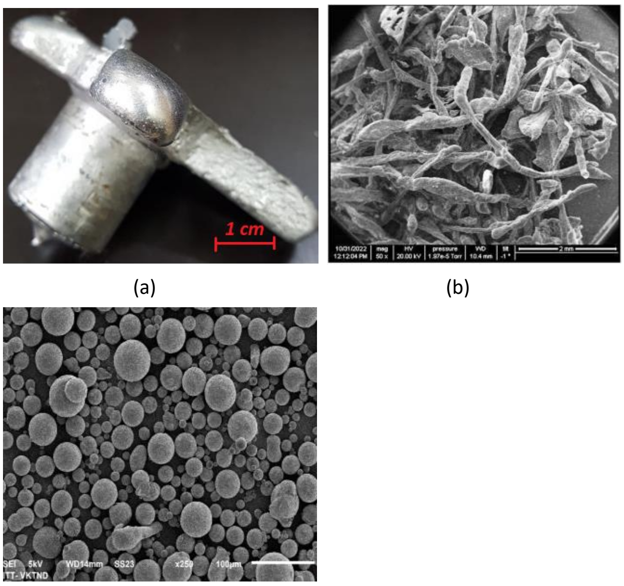
In this study, the criteria to evaluate the influence of these parameters are the morphological structure, particle composition, and the efficiency of the metal powder dispersion process. The methods for evaluating the properties of the output metal powder are listed as follows. *Method to determine the morphological structure of metal particles*: The surface morphology and particle size of fabricated AlSi10Mg aluminum powder were investigated by scanning electron microscopy method (SEM - Scanning Electron Microscope) according to the standard ГОСТ 25849-83. *Method to determine the particle size distribution*: the particle size and its distribution (Particle Size Distribution - PSD) of AlSi10Mg metal powder is determined by two methods: the non-direct measurement method based on standard sieves according to ГОСТ 18318-94 and the practice of direct measurement using particle size analyzer based on laser scattering according to ASTM D4464-15. In addition, the chemical composition of the obtained AlSi10Mg metal powder was determined according to ASTM E1621-21 standard by X-ray fluorescence spectroscopy (XRF).

3 THE RESULT AND ANALYSIS

3.1 *Influence of the liquid metal temperature*

The effect of temperature on the dispersion process was first investigated through the metal powder's morphological structure obtained after fabrication. Meanwhile, the other parameters of the dispersion process were set as liquid metal flow rate at 0.25 kg/min, distance from the top surface of the centrifugal disc to the outlet surface of the gas nozzle *h* = 1.5 cm. The results of SEM imaging of metal powders obtained at different dispersion temperatures are presented in Figure 4.

From the SEM images shown in Figure 4, it is found that at a temperature of 700 °C, the liquid metal has a high viscosity; after pouring it into the hopper, the liquid metal does not flow down the liquid metal pipe, so the dispersion phenomenon cannot occur. (Figure 4a). At a temperature of (770 ÷ 830) °C the metal powder obtained is rod-shaped with a length of up to 2mm (Figure 4.b).



(a) (b)

(c)

Figure 4. SEM images of metal powder obtained at different dispersion temperatures

The reason may be that at this temperature range, the liquid metal has a high viscosity and high surface tension so that the liquid metal is coagulated and crystallized before being dispersed and spherical under the effect of surface tension. When the temperature of the liquid metal is increased, the obtained metal grain profile tends to be gradually globalized. At a temperature of 900 °C, about 99% of metal powder with particle size less than 100 µm has a spherical shape (Figure 4c). This temperature of the liquid metal will be used in further stages.

3.2. Influence of liquid metal flow

In this investigation stage, the other conditions of dispersion were set as:

- Liquid metal temperature when dispersed 900 °C;
- The distance from the top surface of the centrifugal disc to the outlet of the gas nozzle *h* = 1.5 cm;

The survey results show that in the case where the inner diameter of the liquid metal conduit is 6 or 7 mm, about 10 to 12 % of the powder product is with particle size less than 0.4 mm (Figure 5a); from 25 ÷ 27% is in the form of thin flakes, less than 2 mm in size (Figure 5 b); the rest is thin sheets, up to (1 ÷ 2) cm in size (Figure 5 c).

(a) (b) (c)

Figure 5. Images of products of AlSi10Mg liquid alloy dispersion

In case the inner diameter of the needle guide is 3 ÷ 5mm, the number of thin sheets (1 ÷ 2) cm in size is significantly reduced, but the number of small pieces is still relatively large. The amount of metal powder with particle size less than 0.4 mm accounts for 47.3 %, 32.6 %, and 28.4 % of the total weight, respectively. The particle size distribution of the metal powder is relatively similar for all three cases (Figure 6).

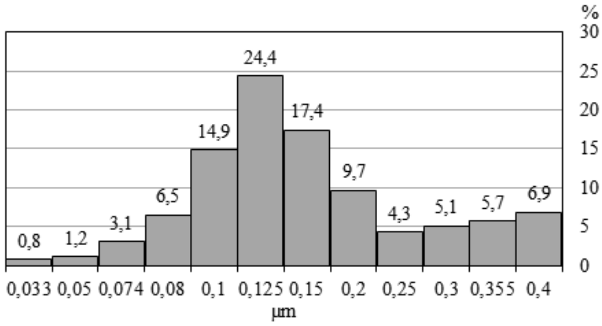
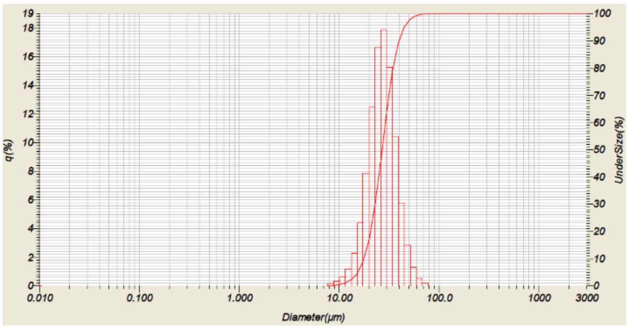
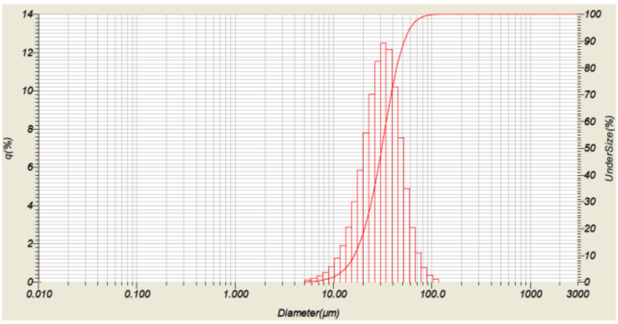


Figure 6. Particle size distribution of metal powder with a size less than 0.4 mm

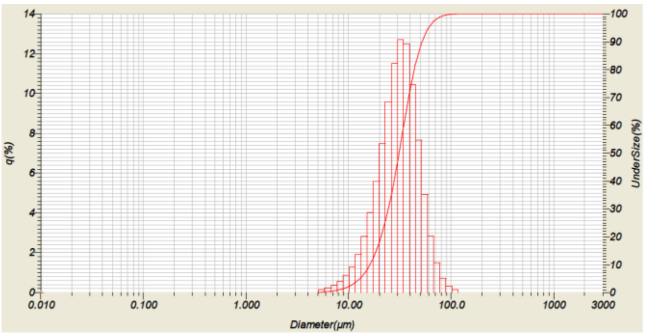
The results of SEM imaging of metal powder with a size of less than 100 µm showed that the metal powder over 90% of the obtained powder has the standard spherical shape. The metal powder content of this group is 11.6 %, 8.3%, and 5.2 % of the total in cases of different inner diameters of the liquid metal conduit of 3 mm, 4mm, and 5mm, respectively. The results of determining the particle size distribution by laser scattering method show that the metal powder group fabricated by liquid metal pipes of 3mm diameter has a narrower particle size distribution and smaller average particle size d50 (Figure 7, 8).



(d = 3 mm)



(d = 4 mm)



(d = 5 mm)

Figure 7. Results of determination of particle size distribution of metal powder group with particle size less than 100 µm

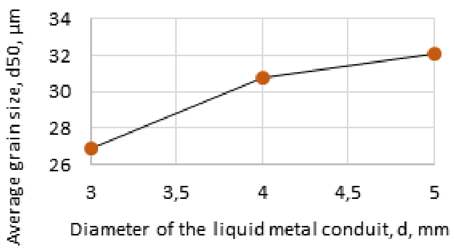


Figure 8. Average particle size dependence on liquid metal conduit diameter

It should be noted that reducing the size of the liquid metal conduit to less than 3 mm to reduce the liquid metal flow did not give satisfactory results. The reason is that the liquid alloy has a rather high viscosity; thus, the surface tension hinders the downward movement of the liquid mass, and the dispersion process cannot take place. The inner diameter of the metal conduit of 3 mm will be used in further stages.

3.3. Influence of the distance from the top surface of the centrifugal disc to the outlet of the gas nozzle

The distance *h* from the top surface of the centrifugal disc to the outlet of the gas nozzle plays a critical role in the dispersion efficiency. The distance h is considered proper when the liquid metal, after being dispersed by the centrifugal rotating disc, will be located where the dispersion gas has the maximum kinetic energy, thereby giving the best dispersion efficiency. Based on simulation results [9], the influence of the distance *h* would be investigated from 0.5 to 3 cm with specific values of 0.5 cm, 1 cm, 1.5 cm, 2 cm, and 3 cm. The other parameters of the dispersion process are set as follows: dispersion temperature is 900 °C, rotational speed of the centrifugal disc is 15 000 rpm; dispersion gas flow and gas pressures are 24 atm and 0.96 m^3/min, respectively; diameter of the liquid metal conduit is 3 mm.

The survey results show that, at the distance *h* = 0.5 cm, the liquid metal droplets, after being dispersed by the centrifugal force caused by the rotating disc, have adhered and gradually accumulated under the inner surface of the gas ring. After about 1.5 min of dispersion, this adhesion and accumulation were so much that the centrifuge disc stopped working (Figure 9).

(a) (b)

Figure 9. Accumulation of liquid metal at the dispersion gas ring *(with h = 0,5 cm)*
(a)– Side view; (b) – Top view

With the distance *h* = 1 cm, the deactivation of the centrifugal disc did not occur. However, a relatively large amount of liquid metal still accumulated on the surface of the dispersion disc and especially on the underside of the heating copper tubes (Figure 10).

Figure 10. Accumulation of liquid metal at the dispersed gas ring and heating copper tube *(h = 1 cm)*

With the distance *h* from 1.5 to 2 cm, the dispersion process gave relatively similar results, with the proportion of metal powder with a size less than 0.4 mm accounting for 44 % to 47 % of the total weight of the dispersed metal. The amount of spherical metal powder, less than 100 µm, accounted for about 10% of the obtained metal powder.

With the distance *h* = 3 cm, the efficiency of the dispersion process has decreased sharply. The proportion of metal powder with a size less than 0.4 mm accounted for only about 21% of the total weight of dispersed metal. The amount of spherical metal powder with a size below 100 µm accounted for about 0.8% of the metal powder obtained. Observing the dispersion chamber, it was found that most of the liquid metal droplets adhered and coagulated on the chamber wall after being dispersed and accelerated by the centrifugal rotating disc. The adhesion position on the chamber wall had a height equivalent to the height of the top surface of the centrifuge turntable. From this, it can be predicted that, at the distance *h* = 3 cm, the kinetic energy of the gas is not strong enough to disperse and change the direction of motion of the liquid metal droplets. As a result, most of the liquid metal, after dispersion by the rotating centrifugal disc, hits the wall of the dispersion chamber (Figure 11).

(a) (b)

Figure 11. The phenomenon of metal adhesion and accumulation on the wall of the dispersion chamber (h = 3 cm)
a – Dispersion chamber; b – Metal plaque coagulated on the chamber wall

3.4. Chemical composition of metal powder after fabrication

The chemical composition of metal powder after fabrication was investigated according to ASTM E1621-21 standard by X-ray fluorescence spectroscopy. The results are shown in Table 3..

Table 3. Chemical composition of metal powder after fabrication.

№	Chemical element	Unit	Quantity
1	Al	%	89,474
2	Si	%	9,133
3	Fe	%	0,264
4	Cu	%	0,0076
5	Mn	%	0,306
6	Mg	%	0,280
7	Zn	%	0,0082
8	Ni	%	0,0148
9	Ti	%	0,101
10	Sn	%	0,0028
11	Pb	%	0,0016
12	Impurity	%	0,410

Comparing the chemical composition of the metal powder after fabrication with the chemical composition of the input material, it was found that the contents of chemical elements in the aluminum powder and in the basic cast aluminum sample were similar. Besides the change in the composition of some elements (due to burning or measurement errors), the biggest limitation is that the impurity content in the powder is increased compared to the input material (0.262% in cast aluminum and 0.41% in aluminum powder). However, the contents of the main elements in the alloy, which are Si, Mg, Mn and Fe, are still within the thresholds in accordance with the prescribed standard on chemical composition of metal 3D printing powder of AlSi10Mg mark.

4 CONCLUSION

In this study, on the basis of experimental survey results, the influence of a few technology parameters was investigated using a set of metal 3D printing powder fabrication equipment researched and constructed by the authors. Those parameters present the main design of equipment and the fabricating process that affects the powder morphological structure, particle size composition, and metal powder dispersion process efficiency. The survey results show that the liquid metal temperature of dispersion plays a decisive role in order to form the grain profile of the metal powder, while the efficiency of the dispersion process is greatly influenced by the liquid metal flow rate. The main technological parameters and structural parameters that have been optimized as follows:

+ Liquid alloy temperature of dispersion: 900 °C;

+ Diameter of liquid metal conduit: 3 mm;

+ Distance from the top of the centrifugal disc to the air outlet of the nozzle: h = 1,5 ÷ 2 cm;

These research results are the foundation for the authors to conduct further studies and surveys to perfect the equipment and technology for manufacturing metal powders oriented for application in 3D printing technology.

FUNDING

This research was funded by Military Institute of Science and Technology of Vietnam with the project code 152/2021.

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