Study on the flowability of TC4 Alloy Powder for 3D Printing

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Abstract: The flowability of metal powder is an important factor that can affect the quality of printing metal parts in powder bed, which has a significant effect on the quality of 3D printed parts. In this paper, the commercial TC4 powder prepared by plasma atomization (PA), Electrode Induction-melting inert Gas Atomization (EIGA) and plasma rotation electrode process (RFP) was used as the research object. The traditional static flow test method and FT4 powder rheometer were used to test the dynamic performance of TC4 powder. The flowability properties of powder materials are characterized by the combination of integral properties and Dynamic rheological properties to determine the more important powder flowability parameters for 3D printing and to provide a theoretical reference for the preliminary screening of powder for printing. The results show that the particle size, particle size distribution, particle surface finish and particle size uniformity have great influence on the flowability, but the effect of particle size composition on the apparent density is not a single value. The powder sample with good surface finish and uniform particle size has good flowability. The basic flow energy of the powder sample produced by PA is small, the shear stress between the powder layers is small, and it is easy to slide，but not easy to improve the flowability of powder by aeration. The basic flow energy of powder samples produced by EIGA is the highest. Powder compressibility is the worst. Its air permeability is not good, the wall friction action is small, is not easy to adhere. The effect of vibration on the flowability of powder is small. The basic flow energy of the powder samples produced by RFP is smaller because of its large particle size and better sphericity. The compressibility and permeability of the powder are better. The shear action between the powder layer and the interlayer and the friction on the wall are smaller and the relative viscosity is weaker. The powder is insensitive to the flow rate, and the effect of vibration on the flowability is relatively small.

1. Introduction

As the main raw material of 3D forming process, metal powder has a significant influence on the quality of 3D printed parts [1]. In actual production, many operations involve the storage, transportation, supply, mixing and filling of powders. The technological properties of powder materials must reach a certain standard. Before they leave the factory, only five technical indexes such as chemical composition analysis, particle size distribution detection, Hall flow rate, vibrating density and apparent packing density should be carried out [2]. However, users often use different batches of metal powder from the same manufacturer or qualified powder from different manufacturers in the process of use, which results in poor performance stability of the printed products. It is difficult to distinguish the difference between samples by traditional methods of particle size distribution, Hall velocity, vibrating density and apparent density.

The properties of powder, such as sphericity and flowability, directly affect the quality of final printing parts in powder bed printing [3]. Therefore, it is very important to evaluate the properties of powder before printing. At present, the flowability of powder is advocated as an important index to measure the technological properties of powder materials [4-5]. As we all know, the flowability of powder materials is affected by many other parameters, such as powder morphology, particle size distribution, vibrational density, apparent density, angle of repose, angle of collapse, angle of plate, etc [6]. Thus, it is very important to choose a method to evaluate the flow performance of powder.

In this paper, the commercial TC4 powder prepared by PA, EIGA and RFP was used as the research object. The traditional static flow test method and FT4 powder rheometer were used to test the dynamic performance of TC4 powder. The flowability properties of powder materials are characterized by the combination of integral properties and shear properties to determine the more important powder flowability parameters for 3D printing and to provide a theoretical reference for the preliminary screening of powder for printing.

2. Experimental materials

In this experiment, three kinds of commercial TC4 powders produced by PA, EIGA and RFP were selected as experimental objects and the sample numbers were listed in Table. 1, besides the main components of these materials were shown in Table 2. The particle size of the powder sample was measured by the Mastersizer 3000 laser particle size analyzer, the surface morphology of the powder was examined by the JSM-5600LV scanning electron microscope (SEM), the apparent density of the powder sample was measured by funnel method and Scotty volumeter method. The flow properties of the samples were tested by FT4 powder rheometer (Freeman Technology, UK). Each sample is tested using the same batch of materials.

Table 1. Manufacturing process of three kinds of Powder samples.

Sample number	Manufacturing process
1#	PA
2#	EIGA
3#	RFP

Table 2. Powder sample composition (Percentage of mass, %).

Sample number	Al	C	Fe	V	Ti	H	O	N
1#	6.52	0.015	0.198	4.01	Bal.	0.0026	0.082	0.018
2#	5.96	0.018	0.120	3.96	Bal.	0.0024	0.102	0.015
3#	6.28	0.011	0.176	4.00	Bal.	0.0039	0.105	0.009

3. Results and analysis

3.1. Static flowability test of powder sample

3.1.1. Hall flow rate and apparent density of Powder samples.

Apparent density is the comprehensive embodiment of various properties of powder, which is very important for powder metallurgy, the stability of mechanical parts production process and the control of product quality [7]. In this paper, the apparent density of powder samples was measured by funnel method and Scotty volumeter method respectively. The experimental results are shown in Figure 1. From the Hall flow rate test results, it can be seen that it takes only 12.0 seconds for 50g of 1# powder sample to flow through the standard funnel with specified pore diameter, indicating that the flowability of the powder sample is the best, 3# sample is slightly worse, and 2# sample is the worst. The same results were obtained for the apparent density by Scott capacity method.

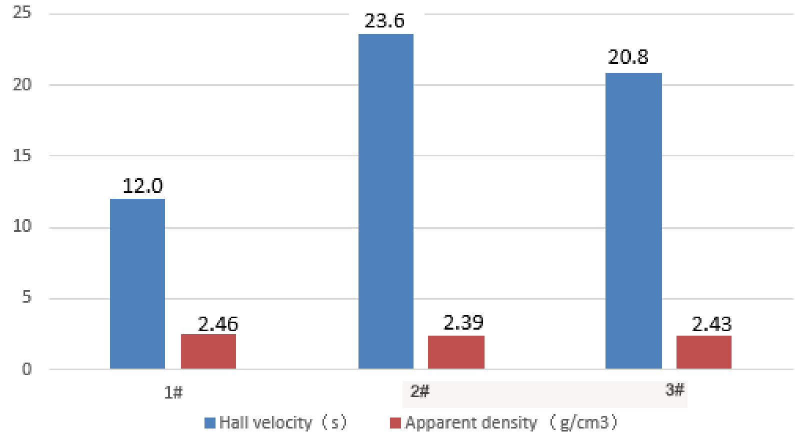

Figure 1. Hall flow rate and apparent density of Powder samples.

3.1.2. Particle size distribution of powder samples

The powder samples were dried in vacuum oven and cooled to room temperature. The particle size of the powder samples was measured by Mastersizer 3000 laser particle size analyzer. The particle size distribution curves of the three kinds of samples were shown in Figure 2. Powder size measurements (D 10 - D 50 -D 90) are shown in Table 3. It can be seen that the particle size of the 1# is smaller, the particle size distribution is the most concentrated, and the uniformity is better than the other samples; the size of sample 2# is the largest, the particle size distribution is wide, and the uniformity is poor; the particle size of sample 3# is relatively large and the distribution is more concentrated. To analyse Figure 1 and 2, the following conclusions can be drawn from the data of Table. 3: The particle size and particle size distribution of the powder sample have great influence on the flowability, but the effect of the particle size composition on the apparent density is not a single value, which is usually determined by the effect of particle filling and bridging. If the former is the main one, the apparent powder density will be increased. In order to obtain the needed density value of powder loose packing, it is feasible to classify and batch reasonably.

Table 3. Results of Particle size Measurement (D10-D50-D90).

Sample number	1#	2#	3#
powder size	21.8-32.8-48.6	20.7-34.1-54.2	24.4-35.9-52.5

3.1.3. Microstructure Analysis of Powder samples

From the SEM photos of the three kinds of powder samples (Figure. 3), it can be seen that the particles of 1#, 2# and 3# powder samples are spherical, and some small satellite spheres are attached to the surface of the large particles. Among them, the particle surface of 1# powder sample is smooth and clean, and the particle size is relatively uniform. There are some gourd-like particles on the surface and a small amount of agglomeration and broken particles on the surface. The particle size of the powder sample is relatively large. The size is uneven. The more regular the surface shape of powder sample, the greater the loose density of powder, and the less irregular the surface shape of powder is, the lower the apparent density is. One of the reasons is that the more irregular the powder, the bigger the specific surface and the greater the friction between particles, so the loose density is lower and the flowability is worse [8-9].

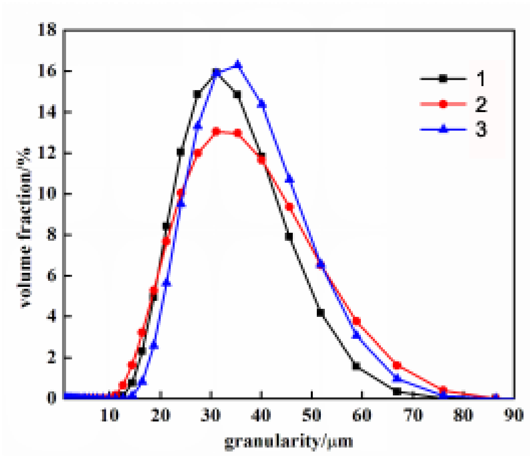

Figure 2. Particle size distribution of different powder samples.

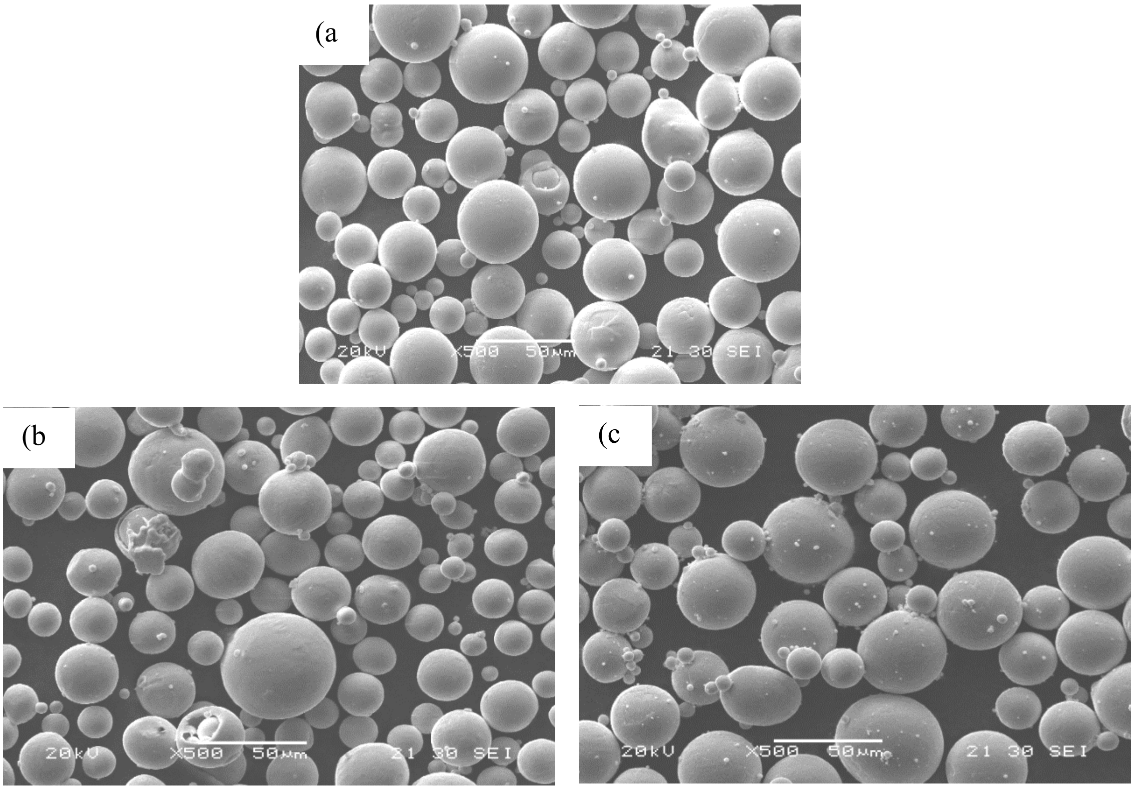

Figure 3. Microstructure of powder sample (a) sample 1# (b) sample 2# (c) sample 3#.

3.2. Dynamic flowability testing of Powder samples

3.2.1. Dynamic testing: stability and variable flow rate

The test of stability and variable flow rate means that the blade passes through the pretreated sample powder at a constant or varying rate, and the resistance of the blade is measured to evaluate the flowability of the powder under the free surface. In the process of co The more regular the surface shape of powder sample, the better the flowability, the smaller the basic flow energy of powder sample produced by plasma atomization process, the easier to agitate dynamically, the less flowability can be improved by aeration, the smaller the shear stress between powder layers is and the easier it is to slide. unterclockwise downward movement, the radial torque and axial force acting on the blade are measured, and the energy conversion is carried out in combination with the action distance. The resistance of the powder to the blade during the flow can be obtained, which is expressed by the flow energy [10]. The blade velocity of the first 7 times was tested at a constant speed, and the stability of the powder flow was tested. The velocity of the blade decreased gradually in the following four times, and the sensitivity of the flowability of the powder to the velocity was tested. The test results are shown in Table 4.

Table 4. Dynamic performance of the three powder samples.

dynamic properties	1#	2#	3#
Basic Flowability Energy, BFE (mJ)	335.30	335.30	339.09
Stability Index, SI	1.05	1.02	0.98
Flow Rate Index, FRI	1.24	1.25	1.11
Specific Energy, SE (mJ/g)	2.63	2.45	2.39

The basic flowability energy (BFE) is the flow energy measured through the powder for the seventh time, reflecting the specific properties of the sample. It is generally considered that the powder with good flowability has lower BFE value. From the BFE values of the three kinds of samples, it can be seen that the basic flow energy of 1# and 3# samples is close and small, and the energy needed in the process of dynamic flowing is smaller. The basic flow energy of the 2# sample is higher.

The stability index (SI) refers to the ratio of the seventh to the first flow energy of the blade, which is used to describe the variation of the flow energy of the sample with repeated test. The closer the SI value is to 1, the better the flow stability of the sample is. The SI values of the three samples were close to 1, and the samples were stable during stirring.

The flow rate index (FRI) is the ratio of the 11th flow energy measurement value to the eighth flow energy measurement value. It reflects the sensitivity of the powder to the flow rate. The closer the FRI value is to 1, the more insensitive the powder is to the change of flow rate, and the more stable its flowability is. The FRI values of samples 1# and 2# are higher and sensitive to the flow rate and easily affected by the stirring speed. The FRI of sample 3# is smaller and less sensitive to the flow rate.

The specific energy (SE) is the energy needed to move the powder per unit mass when the blade moves upward, which indicates the flowability of the powder in unrestrained or low stress state, and mainly reflects the adhesion between particles. For example, the specific flow energy of friction and mechanical bite force [11]. 1# sample is slightly larger, and the effect of friction and mechanical interlocking is relatively strong. The specific energy of sample 1 is slightly larger, the friction and mechanical interlocking is relatively strong, and the specific energy of sample 2# and sample 3# is close and small.

3.2.2. Aeration test

The air in the powder has a significant influence on the flow properties.The aeration test is to measure the difference in the flowability of the powder samples before and after the introduction of the air. This test provides an index of the adhesion strength to characterize the mixing, aerodynamic and fluidization of the powder. Generally, the absolute adhesion of the lower inflatable energy represents a smaller degree of absolute adhesion. Generally, lower aeration energy represents less absolute adhesion of powder.

In the experimental process, with the filling of 0-10mm/s air, the powder particles are separated gradually, and the interaction force decreases. That is, the flow energy gradually decreases, finally reaches the steady state, the basic flow energy does not change. The aeration test results of the three powder samples are shown in Fig. 4.The three kinds of powder samples reach stable state under the air of 10mm/s. The aeration energy of the 1 # powder sample is large in steady state, and the powder has a certain viscosity. The aeration energies of 4 # and 6 # samples are close to each other in stable state.

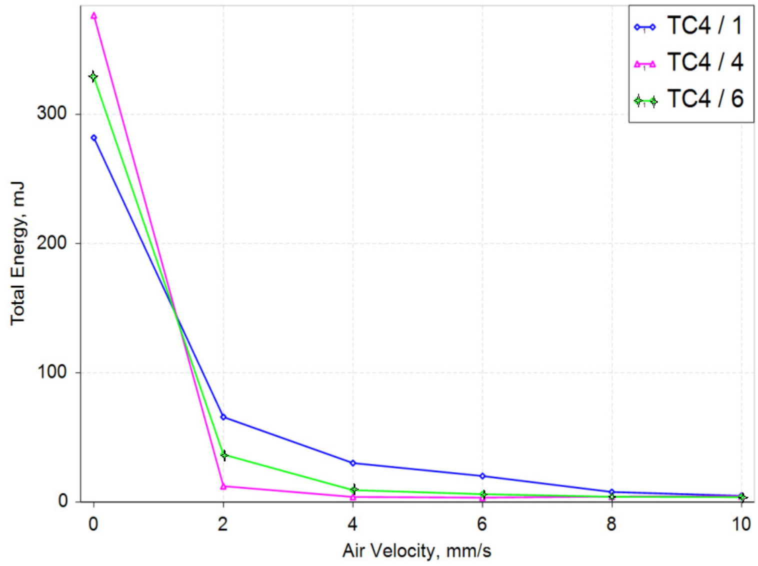

Figure 4. Difference of sample flowability before and after introduction of air.

3.3. Integrity testing

3.3.1. Compressibility Test

Compressibility test is testing the change of volume of sample under different normal stress and this experiment reflects the overall performance of the sample [12]. The results show that the powder is compacted under a certain normal stress, and the volume change caused by storage, transportation, direct pressure and rolling is measured. The three kinds of powder samples were cut after pretreatment, and then the sample was subjected to different normal stress by a breathable piston, and the sample volume was measured to obtain the sample compressibility curve, as shown in Fig. 5.

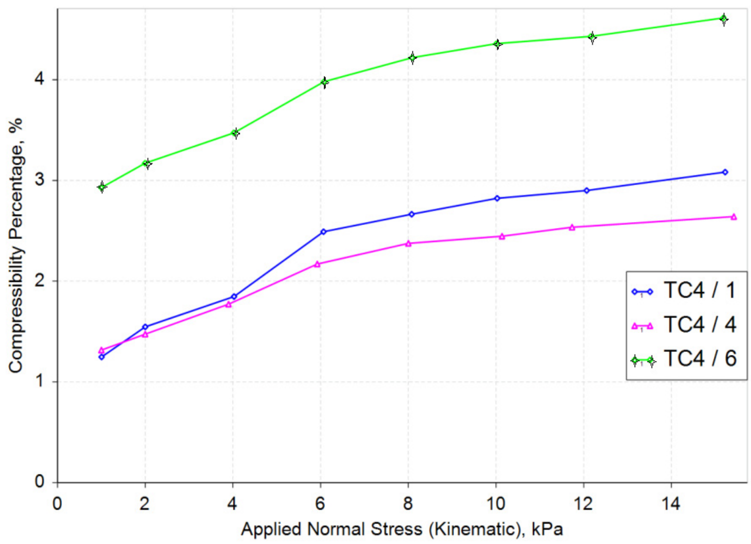

Figure 5. The curves of compressibility.

From Fig. 5, it can be seen that under the condition of increasing stress, the volume of the sample is gradually reduced, the compressibility of the sample is gradually increased, and there is a certain response to the increasing stress condition. At the pressure of 15 KPa, the compressibility of the samples ranged from 2.64% to 4.61%, and the compressibility of sample 1# was close to that of sample 2#, and the compressibility of sample 3# was the largest. When the powder flows in the hopper of a 3D printing device, the powder at the bottom of the hopper gets more force with the amount of powder increasing, so the gap between the powder particles is reduced, and then the bonding force between the powder particles will increase. Eventually, the powder becomes difficult to slide. The powder with large compressibility is easily blocked at the outlet to influence the normal powder extraction of the powder extractor, resulting in the phenomenon of uneven powder spreading and even the failure of the powder laying, which ultimately affects the normal process of printing. With the change of normal stress, the smaller the compressibility of powder is, the better the stacking condition of powder is, the more stable the powder structure is, and the better the uniformity of powder is when printing.

3.3.2. Permeability test

The gas permeability test is based on the compressibility test to fill the powder with a certain rate of gas (2 mm/s), and to measure the pressure drop of the gas passing through the powder with the change of normal stress. The greater the pressure drop, the worse the air permeability of the powder, and the better the air permeability. The permeability test results of the three samples are shown in Figure. 6.

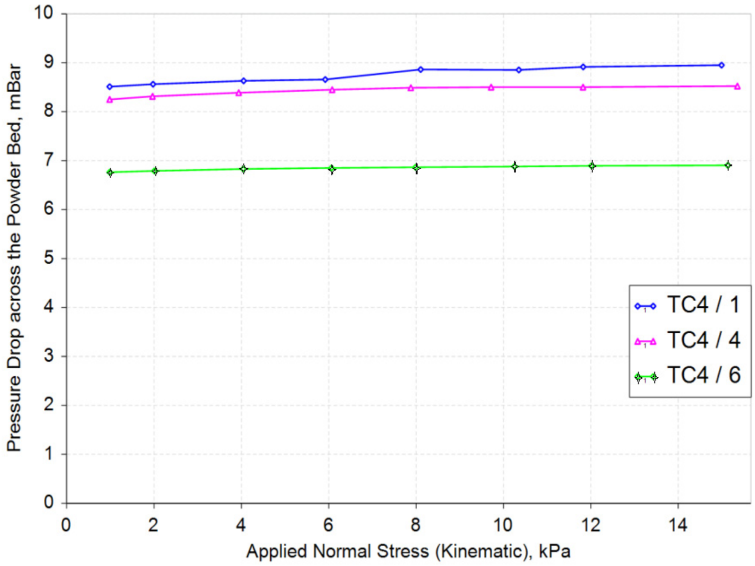

Figure 6. Pressure drop across the powder bedwhen air velocity is 2 mm/s.

Under the increasing stress, the pressure drop of the three samples is stable and the variation range is small. By comparing the pressure drop values under 15 KPa stress conditions, it can be seen that the pressure drop of samples 1# and 2# is larger, the internal air passage is less, the air flow is not easy to pass through the powder, and the permeability is poor, which also verifies the larger viscosity. The permeability of sample 3# is better. The air permeability is mainly related to the particle size and shape of the material, and the air permeability of the powder material with good flowability is not necessarily good. The air permeability mainly affects the powder flowing out from the closed container. The external gas can enter the container through the powder and balance the pressure inside and outside of the container, so that the powder can flow out smoothly. Therefore, the permeability can affect the flowability of the powder under certain conditions, but the powder depends on the gravity drop when the powder bed is printed, the permeability is not the main factor.

4. Conclusion

The flow behaviour of powder is very complex during the 3D printing process, and the flowability of powder can not be evaluated by a single index. In this paper, powder materials were characterized by particle size detection, surface morphology analysis, static flowability test and dynamic flowability test, and the flowability properties of three kinds of powder samples were comprehensively evaluated. The effects of different production processes are compared. The conclusions are as follows:

1) The particle size and particle size distribution of the powder sample have great influence on its flowability. The particle size and particle size distribution of powder sample have great influence on its flowability. The flowability of powder with larger particle size is better. The flowability of the powder with larger particle size distribution range is bad.

2) The surface finish and particle size uniformity of the powder sample have great influence on its flowability. The powder sample with good surface finish and uniform particle size has good flowability.

3) The basic flow energy of powder samples produced by EIGA is the highest. Powder compressibility is the worst. Its air permeability is not good, the wall friction action is small, is not easy to adhere. The effect of vibration on the flowability of powder is small. The basic flow energy of the powder samples produced by RFP is smaller because of its large particle size and better sphericity. The compressibility and permeability of the powder are better. The shear action between the powder layer and the interlayer and the friction on the wall are smaller and the relative viscosity is weaker. The powder is insensitive to the flow rate, and the effect of vibration on the flowability is relatively small.

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