Analysis of Rheological Properties of Selective Laser Melting 17-4PH Powder

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***Abstract*—In the selective laser melting process, high mechanical stability, excellent thermal performance and rheological property are required for printable materials, and due to the change of environmental conditions such as external force and temperature rise during the forming process and the using process of the forming part, The multi-phase structure of the material is changed, so that the mechanical property of the material is changed, and the change of the forming part is mainly manifested in the change of the rheological property. The rheological property of the 17-4PH powder material was studied to ensure the mechanical properties of the material after laser sintering. The results show that the viscosity of the 17-4PH powder material decreases with the increase of the shear rate, has good flow formability during the subsequent selective laser sintering process, And the forming part can meet the mechanical property requirement in the actual production and processing use.**

Keywords-3D Printing; Selective Laser Melting; Metal Rheology; Viscosity; Mechanical Properties

I. INTRODUCTION

Selective laser melting forming ((Selective Laser Melting, SLM) is a rapid development of metal parts precision material increasing manufacturing technology in recent years. It is based on layered manufacturing and layer by layer superimposed forming principle, based on three-dimensional CAD model and metal powder, does not need special die and fixture, and adopts high power density laser to melt metal powder point by point, line by line and layer by layer, so as to obtain high performance and high density metal parts. At present, SLM technology has been used in the preparation of stainless steel, titanium alloy, superalloy and aluminum alloy complex parts[1-5].

17-4PH is generally used in the manufacture of shafts, steam turbines, offshore platforms, helicopter decks and other mechanical components because of its high strength, hardness and corrosion resistance. This material is also widely used in the manufacture and production of turbine blades in aerospace. In the process of SLM molding, the sintered parts are easy to warping and deformed, and the powder sintered parts will show different shrinkage and spheroidizing phenomena, which will affect the accuracy of the parts. Therefore, in order to obtain the molding parts with reduced size and high precision, it is necessary to characterize the Rheological properties of sintered materials[6-8].

There have been a few studies on SLM forming 17-4PH stainless steel at home and abroad. Gu et al.[9] and Spierings et al. [10] have studied the effects of process parameters on pores, microstructure and mechanical properties, Murr et al. [11] have studied the effects of different atmosphere conditions on formed martensite and Austenite, LeBrun et al have studied the effects of heat treatment on mechanical properties[12], and LeBrun et al have studied the effects of heat treatment on mechanical properties. Hu et al studied in detail the pore formation mechanism and characteristics of 17-4PH stainless steel formed by SLM[13].

II. TEST

A. Test materials and methods

1) Rheological part

The 17-4PH powder material with particle size range of 20 ~ 65 μm and average particle size of 30 ~ 40 μm was used in the experiment. The SEM scan of the powder material is shown in FIG.1.

Sample Preparation: A suspension of a 17-4PH powder (FIG.2 (a)) was first prepared for rheological property testing of the material. 50 ml of deionized water (FIG.2 (b)) was prepared in a beaker,8 g of a powder material was added to the de-ionized water, and the resulting suspension was mixed with a stirrer for 2 min; then 0.104 g of hydroxypropyl methylcellulose (HPMC,4000 viscosity, FIG.2 (c)) were added and mixed continuously; after 1 h, 0.136 g of polyethyleneimine (PEI, Mw = 1800, FIG.2 (d) is a bridge-linked flocculation and is continuously mixed until the mixture is homogeneous, and finally a 17-4PH powder suspension mixed sample for the rheological property measurement is obtained (FIG.2 (e)).

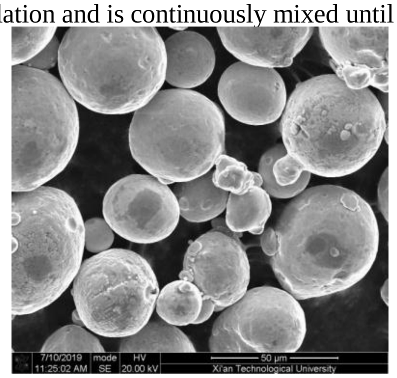

Figure 1. Micromorphology of 17-4PH powder

Figure 2. Material and sample (a) 17-4PH powder material; (b) hydroxypropyl methyl cellulose; (c) polyethylene imine; (d) mixing equipment; (e) mixture sample

2) Trial section

The 17-4PH metal material powder subjected to the rheology test described above was subjected to a test process on a selective laser melting molding apparatus.

3) Mechanical properties analysis part

In this paper, the tensile test of the molding part is carried out, and the stress-strain relationship of the sample under external force load is analyzed, and the yield limit, strength limit, extensibility and section shrinkage of the material can be obtained.

B. *Equipment and instruments*

In this experiment, the viscosity of 17-4PH material was measured by Antonpa MCR301 rotating rheometer, the test temperature was 85 ℃, and the shear rate was 10-150 mm • min.

In the process of trial processing, Huashugaoke FS271M model equipment is selected, the thickness of powder can be adjusted by 0.02~0.1mm, and the scanning speed can reach 15.2 m / s.

TABLE I. VISCOSITY OF THE MATERIAL AT DIFFERENT SHEAR RATES (PA • S)

Rate of shear/s-1	12.82	24.25	35.63	47.04	58.62	70.09	81.46	98.53	115.7
viscosity/ Pa • s	5.353	4.173	3.083	2.484	2.11	1.696	1.464	1.247	1.122

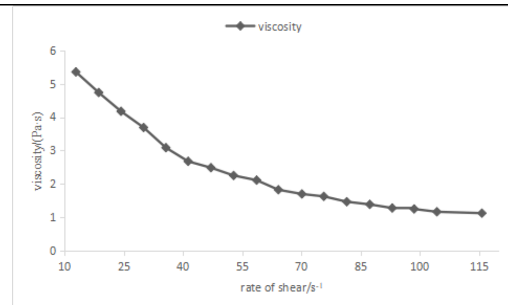

Figure 3. Variation curve of viscosity with shear rate

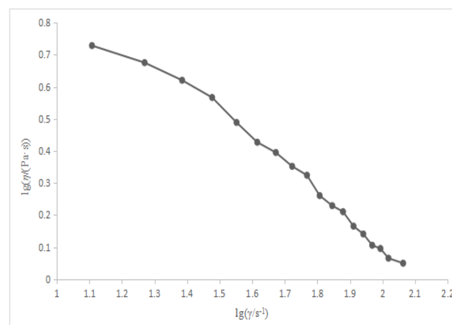

Figure 4. Lgη-lgγ relation curve

The mechanical properties were tested by Shenzhen Sansi CMT5304-30kN electronic universal testing machine, the maximum test force was 300kN, and the deformation measurement range was 0.4-100% FS.

III. RESULTS AND DISCUSSIONS

A. *Viscosity of samples*

In the process of selective laser sintering, the viscosity of the sample is the basic index to evaluate the Rheological properties of sintered materials. The viscosity of the sample can characterize the flowability of the material, that is, the lower the viscosity of the material is, the better the flowability of the material is [15].

Table 1 is the viscosity of samples measured at different shear rates at constant temperature by rotating rheometer. It can be seen that with the increase of shear rate, the viscosity of the sample shows a downward trend, which reflects the shear viscosity reduction characteristics of the material. In this process, 17-4PH metal powder materials show obvious pseudo-plastic flow. The curve of viscosity with shear rate is obtained, as shown in figure 3.

TABLE II. CHEMICAL COMPOSITION OF 17-4PH METAL POWDER (MASS FRACTION,%)

Ingredient	C	Mn	Si	Cr	Ni	Cu	Fe
Content	≤0.07	≤1.00	≤1.00	15.5～17.5	3.0～5.0	3.0～5.0	all

TABLE III. PROCESS PARAMETERS OF SELECTIVE SINTERING

Parameter	sinter point/(℃)	powder spreading thickness/(mm)	scanning speed/(mm/s)	laser frequency/(W)
Value	1300	0.05	2000	500

The system viscosity of metal powder material can reflect the uniformity of powder particle mixing with its binder, that is to say, under the same mixing condition and the same powder loading quantity, the smaller the viscosity of metal powder system is, the better the compatibility between powder particle and binder system is, the more uniform the mixture is, the better the liquidity is. It can also be well formed in the process of selective laser melting.

By analyzing the viscosity of the sample at different shear rates, it can be seen that with the increase of the shear rate, the viscosity of the sample shows a downward trend, which reflects the shear viscosity reduction characteristics of the material. In this process, the 17-4PH metal powder material shows obvious pseudo-plastic flow. In addition, when the shear rate is low, the apparent viscosity of the sample changes dramatically, while when the shear rate changes in the range of higher value, the apparent viscosity of the sample changes little. This is mainly due to the ordering between powder particles and the equalization of adhesive molecules, and when the high shear effect is received, the long molecular chain in the adhesive system is oriented along the flow direction, which makes the arrangement orderly. At the same time, the metal powder particles consume energy in the process of agglomeration, which will lead to the decrease of the viscosity of the sample.

In a selective laser-melting molding system, the metal powder material generally exhibits pseudo-plastic rheological behavior. for a plastic fluid material, there are:

$$\eta = K\gamma^{n-1} \tag{1}$$

In the formula, η is the viscosity, γ is the shear rate, K is the coefficient, n is the fluidity index, and the value of n <1. n represents the sensitivity of the fluid to the shear rate, which plays an important role in the forming of the metal powder material. In general, the higher the value of n, the slower the viscosity of the material with the rate of change of the shear rate, at which point the flow deformation stability of the powder material is good, but the metal powder material in this case does not exhibit sufficient shear thinning behavior. On the basis of this, it is proposed that the value of n is as small as possible on the premise of n> 0.2[16]. This value is because, as the n value decreases, the faster the viscosity of the powder material changes with the change of the shear rate as the n value decreases. Selective laser melting is performed rapidly at a temperature, so this

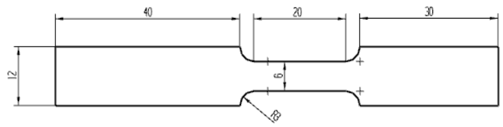

Figure 5. Design dimensions and molding parts of sample parts for trial processing

property of the material facilitates the formation of the material.

According to formula (1), the double logarithmic diagram of viscosity and shear rate is made, that is, the relationship curve of lgη-lgγ, as shown in figure 4.

According to the logarithmic slope of the logarithmic relationship between the viscosity of the material and the shear rate in Fig. 3, we can calculate the value of the epidemic index n of the sample, n=0.2898.

In conclusion, 174PH metal powder material has good fluidity and shear viscosity reduction property, and has good flow forming performance in selective laser melting process.

B. *Selective laser melting test molding*

Selective laser melting of 14-4PH metal powder materials is carried out, and the powder materials and components are shown in Table Ⅱ.

The technological parameters of laser melting are shown in Table Ⅲ.

The design size of selective laser sintering test melting using 17-4PH metal powder and the sample after forming are shown in Fig.5.

C. *Mechanical tensile test*

The flow behavior of the object will also occur under the action of external force, which is manifested in the mechanical properties of the material. For the selective laser melting molding parts, whether it can have good mechanical properties to better play a role in the subsequent actual production process is very important.

In this paper, the tensile test of selective laser melting parts with 17-4PH metal powder at room temperature is carried out.

In this part, the relationship between time-varying stress and strain during deformation is analyzed by axial tensile test of selective laser melting parts, and the relationship between stress and strain is analyzed and calculated.

The tensile tests at room temperature and static load are carried out on the material testing machine. The stress-strain relationship of the material samples can be obtained in the process of analysis. We find that the stress-strain relationship of SLM molded parts during tensile test will occur in the process of tensile deformation of samples, and the following four stages of deformation will occur in the process of tensile deformation of samples.

1) In the first stage-elastic deformation stage

when the stress σ and strain ε of the material are in a positive proportional relationship, the maximum stress that the specimen can bear is σ_{nu} called the proportional limit [17].

TABLE IV. FORMULA FOR CALCULATING MECHANICAL PROPERTIES OF MATERIALS

Performance index	Yield strength σs	Tensile strength σB	Elongation at break δ	Reduction of area Ψ
Computational formula	$\sigma_s = \frac{F_s}{A}$	$\sigma_B = \frac{F_B}{A}$	$\delta = \frac{l_1 - l_0}{l_0} \times 100\%$	$\Psi = \frac{A_0 - A_1}{A_0} \times 100\%$

l_1 is the length of specimen deformation, and l_0 is the length before the test. A_0 is the cross-sectional area of the sample before the test, and A_1 is the minimum cross-sectional area after necking.

At this time, the deformation mode of the material follows Hook's law, $\sigma=E\varepsilon$, where E is the tensile elastic modulus. At this stage, if the load force loaded on the sample is unloaded, the material will return to its original state.

2) In the second stage - the yield (flow) stage

Although the external force fluctuates in a small range, the deformation increases significantly when the material receives the external force for deformation. That is to say, at this stage, the material temporarily loses its ability to resist deformation, and the corresponding stress value becomes the yield limit of the material σ_s . If unloaded at this time, the material will not return to the original state. Therefore, the yield strength of the material determines the mechanical properties of the material to a great extent.

3) The third stage-the strengthening stage

After the yield stage, the material recovers the ability to resist deformation, which is called strengthening. At this stage, the highest point of the curve corresponds to the stress called the strength limit of the material σ_B . The strength limit is the maximum stress that the metal specimen can bear in the whole tensile test.

4) In the fourth stage-necking failure stage test

After the stress of the specimen has gone through the strengthening stage, the direct decrease of a certain local position of the specimen suddenly becomes small, and the deformation is the necking of the material at this time. After that, the axial deformation of the specimen is mainly concentrated at the necking point.

According to a series of formulas given in Table Ⅳ, the yield strength is 1163 MPa, the tensile strength is 1357 MPa, the elongation after break is 17%, and the section shrinkage is 45%. It can be found that the 17-4PH metal powder material has strong mechanical properties after selective laser melting.

IV. CONCLUSION

In the light of the characteristics of high-temperature melting, solidification and recrystallization of the material in the selective laser melting process, and the characteristics of ensuring that the processed forming part can meet the actual using requirement of the workpiece, the metal rheological property of the selective laser sintering material is researched, and the mechanical property of the molded part is analyzed and tested, and finally the following conclusion is obtained:

- Carrying out the metal rheological property analysis on the 17-4PH metal powder material which is added with different binder under different temperature environment, It is found that the 17-4PH metal powder material exhibits linear shear-viscosity characteristics at different shear rates at a constant temperature, and the flow properties of the 17-4PH powder material can be the best.
- Performing selective laser sintering and melting processing test on the 17-4PH metal powder, and the processed sample piece is a molded part capable of directly performing mechanical property analysis.
- Carrying out axial tensile test on the molded part, and analyzing the stress and strain of the stress state of the sample piece during the loading process, The formed part obtained by the selective laser sintering of the 17-4PH metal powder material has the requirements of the mechanical property required in actual working production.

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