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ARTICLE

Quantitative Characterization of Superalloy Powder Particle Shape Using Dynamic Image Analysis Technique

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Abstract: Eight shape parameters were selected to qualitatively and quantitatively characterize the particle shape of superalloy powders using field emission scanning electron microscopy (FE-SEM) and dynamic image analysis (DIA) technique. The results show that Krumbein sphericity (SPHT_K), aspect ratio (AR), compactness (Compct) and Krumbein roundness (RDNS_C) demonstrate a significant distinction degree of superalloy powder particle shape, and can be used as critical parameters for the characterization of particle shape. In addition, the distinction degree of circularity (C), sphericity (SPHT) and SPHT_K increases gradually. In particular, the SPHT_K has the highest distinction degree among the three shape parameters. By comparing the difference in distinction degree among eight shape parameters, it is found that the distinction degree of circularity (C), sphericity (SPHT), convexity (Conv_A) and solidity (Solid) is not significant.Therefore, the SPHT_K, AR, Compct and RDNS_C are selected as the shape parameter combination to quantitatively characterize the particle shape. The combination of these four shape parameters can be used to quantitatively characterize the change of superalloy powder particle shape. Meanwhile, it provides a data basis to effectively optimize the atomization processing parameters. The promotion of the development of additive manufacturing and advanced powder metallurgy technology in future was discussed in the end.

Key words: superalloy powders; particle shape; quantitative characterization; dynamic image analysis

As essential raw materials, superalloy powders are widely applied in 3D printing and advanced powder metallurgy technology. The research related to these technologies is undergoing rapid progress[1-3]. According to relevant standards, superalloy powders should have spherical particle shape. Many researchers indicate that the particle shape is an important characteristic which determines distinctive properties of the powders[4,5]. For example, the particle shape directly impacts the flowability and bulk density of the powders. The mechanical properties of alloys are likely to be affected by particle shape. Nam et al[6] found that the differences in particle shape markedly affect the microstructures and mechanical properties of the alloy. These results are attributed to the spherical powders forming fine and uniform microstructures after sintering. Gülsoy et al[7] investigated the influence of particle shape on mechanical properties of injection molded Ti alloy powder. The results show that the irregular shape powder has lower mechanical properties than spherical shape powder. Miyake et al[8] revealed that the compressive strength of sintered porous alumina is much higher for the spherical particles than for the rod-like and disk-like particles. Thus, it becomes a major issue to quantitatively analyze the superalloy particle shape for optimizing the mechanical properties of alloys.

The shape parameters which can be used to characterize particle shape have been widely employed in materials science[9,10] and food science[11,12]. Particle shape is an envelope formed by all the points on the surface of the particle. It is generally characterized by two-dimensional in-plane image projections of a three-dimensional particle. A specific shape feature of a particle is quantitatively described by a numerical value which is called the shape parameter. Sergio et al[13] investigated the powder morphology of wheat flour using four shape factors, including the circularity, elongation,

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compactness and roughness. The results show that small particles have a more regular shape than the large particles. Saad et al[14] collected five kinds of microcrystalline cellulose pellet samples at different time intervals during deformation process, suggesting that the particle shape is closely related to its physical properties.

A considerable amount of research characterizing the particle shape of non-metallic particles using DIA has been published. Otilia et al[15] studied coal particles through aspect ratio and circularity. The results suggested that the heat and mass transfer characteristic are strongly influenced by coal particles shape. The particle shape of talc mineral ground by various types of mills is then determined through the dynamic image analysis. Thus, the result of DIA can be applied for quality control of the products of talc mineral[16]. Research was also conducted on the determination of the particle size distribution of different coals ground by various mills, following the comparison of mechanical sieving and DIA. Thus, DIA can be recommended for particle size distribution analysis of fine particulate coals, minerals, and similar products[17]. The shape analysis of barite mineral ground by various mills was conducted by the DIA technique. The shape differences were quantified by creating different grinding in terms of the most common shape parameters, referring to as the aspect ratio and circularity. The research proposed that DIA can be used as a tool for the characterization of barite particles in some industries, where particular shape and morphology of barite are required[18,19].

In summary, the DIA is mainly used to measure the rock and mineral particles. However, few studies have addressed the quantitative characterization of metal powder through DIA. The current method which is widely recognized to characterize the powder shape is field emission scanning electron microscopy (FE-SEM). This method is time consuming and is not able to characterize the particle shape quantitatively. The aim of this study is to control the particle shape in a more accurate manner and to provide the specific shape powder for 3D printing and advanced powder metallurgy technology. Therefore, the DIA technique is used for quantitative characterization of superalloy particle shape in this study.

1 Materials and Methods

Three kinds of superalloy powders in different shapes were prepared by argon atomization (AA). The samples were named as 1#, 2# and 3#. The powders were mechanically sieved with a standard vibrating sieve. Then the powders with particle size ranges of 53~60, 45~53, 38~45, 25~38 μm, and less than 25 μm were obtained subsequently. The powder shape was qualitatively characterized by field emission scanning electron microscopy (FE-SEM). The differences in the shape of three powders were compared.

In order to quantitatively characterize the difference of powder shape, the Camsizer X2 dynamic image analysis (DIA) developed by Retsch technology was employed. The compressed air dispersion module was applied to fully disperse the powder particles. Meanwhile, two high-frequency camera lenses with 4.2 million pixels were employed to capture and photograph the powder particles. Each sample was tested for 3 to 5 min, and more than ten million of particles can be detected. Afterwards, the Camsizer X2 analysis software was used to process the obtained images. The powder shape characterization parameters were calculated and obtained. In this study, eight shape characterization parameters, SPHT_K, AR, Compct, RDNS_C, Conv_A, Solid, C and SPHT were selected to quantitatively characterize the powder shape. Then the original data was exported from the Camsizer X2 software to the Origin to clearly illustrate the results.

2 Definition and Algorithm of Shape Parameters

In this study, eight shape parameters were used to quantitatively characterize the particle shape. The following description are the definition and algorithm of the eight shape parameters:

Krumbein derived an equation for estimating Wadell's sphericity from measurement of three orthogonal axes of a particle. Krumbein intercept sphericity (Ψ_{int}) is an important 3D macroshape descriptor which can more accurately characterize the particle shape than 2D shape descriptor. Its value is obtained by the long axis dimensions a, intermediate axis dimensions b and short axis dimensions c. The short axis dimensions c is determined by the vertical plane perpendicular to the maximum projection area on horizontal projection plane. The principal assumption is that the rock particle approximates an ellipsoid, and Krumbein intercept sphericity (Ψ_{int}) describes the volume ratio of the ellipsoid defined by the three axes to the circumscribing sphere[20,21] (Fig.1a).

$$\Psi_{\text{int}} = \sqrt[3]{\frac{(\pi/6)abc}{(\pi/6)a^3}} = \sqrt[3]{\frac{bc}{a^2}} \tag{1}$$

As the shape parameters obtained by DIA are based on two-dimensional projection images, the Krumbein sphericity is related to the proportion between Feret diameter 1 ($X_{\text{Fe},1}$) and Feret diameter 2 ($X_{\text{Fe},2}$) of the images. The $X_{\text{Fe},1}$ and $X_{\text{Fe},2}$ are mutually perpendicular. Thus, the Krumbein sphericity (SPHT_K) is defined as follows (Fig.1b).

$$\text{SPHT\_K} = \min(X_{\text{Fe},1}/X_{\text{Fe},2}) \tag{2}$$

The aspect ratio (AR) is a widely used macroshape descriptor describing the relationship between minimum Feret diameter $X_{\text{Fe min}}$ and maximum Feret diameter $X_{\text{Fe max}}$. The particle elongation is related to the ratio $X_{\text{Fe min}}/X_{\text{Fe max}}$. For a spherical particle, AR is equal to 1, the higher the particle elongation, the smaller the ratio of AR[22] (Fig.1c).

$$\text{AR} = \frac{X_{\text{Fe min}}}{X_{\text{Fe max}}} \tag{3}$$

Compactness (Compct) is a macroshape descriptor which displays the degree of a particle similar to a circle, considering the overall form of the particle. The compactness is determined by the $X_{\text{Fe max}}$ and the particle projection area A[23] (Fig.1d).

$$\text{Compct} = \frac{\sqrt{\frac{4A}{\pi}}}{X_{\text{Fe max}}} \tag{4}$$

Krumbein roundness (RDNS_C) is a critical shape parameter. This index is used to quantify the sharpness of particle corners. It was first distinguished from sphericity by Wadell[24]. Using two-dimensional projections of particles, Wadell defined roundness as the ratio of the average curvature radius of particle corners (r_i) to the radius of the maximum inscribed circle ($r_{\text{max-in}}$) [25,26]. The algorithm of RDNS_C is illustrated as follows[27,28] (Fig.1e):

$$\text{RDNS\_C} = \frac{\sum (r_i/N)}{r_{\text{max - in}}} \tag{5}$$

Circularity (C) is a mesoshape descriptor defined as the degree that the particle of its projection area is similar to a circle. It is sensitive to both overall shape and spikiness. It indicates the similarity degree of a particle to a disc, in terms of the smoothness of the perimeter *P*. It is a similar parameter to sphericity which is determined by the particle projection area *A* and the corresponding particle projection perimeter *P*[29] (Fig.1d).

$$\text{C} = \sqrt{\frac{4\pi A}{P^2}} \tag{6}$$

Sphericity (SPHT) is a mesoshape parameter which is frequently used to describe the degree that the particle or its projection area approaches to a circle. As an alternative approach to measure circularity, SPHT is frequently used. Because its equation has a squared term, this parameter is more sensitive to subtle variations in the area-to-perimeter ratio than circularity. For this reason, this shape factor is also known as high sensitivity (HS) sphericity. The sphericity is influenced by both overall form and spikiness. Sphericity is an index described as "similarity to a perfect circle", i. e., a perfect circle has a sphericity of 1 while a spiky or an irregular object has a sphericity value closer to 0. SPHT is described in Eq.(7)[30-33] (Fig.1d):

$$\text{SPHT} = \frac{4\pi A}{P^2} \tag{7}$$

Convexity (Conv_A) is a mesoshape descriptor to describe the convexity degree of a particle. It is usually defined as the square root of projection area of a real particle (*A*) divided by the area of the convex hull bonding the particle ($A_{\text{conv}}=A+M+N$) [34](Fig.1f):

$$\text{Conv\_A} = \sqrt{\frac{A}{A_{\text{conv}}}} \tag{8}$$

Solidity (Solid) displays the convexity, determined from the measured particle area *A* and the convex particle area $A_{\text{conv}}=A+M+N$. This shape parameter characterizes the overall convexity of a particle. Because of the difficulty of conducting three-dimensional shape analysis, it is evaluated from the 2D projection of the particle, as illustrated in Fig.1f. The Solid is defined as the ratio of the actual projection area and the convex hull area[35-37].

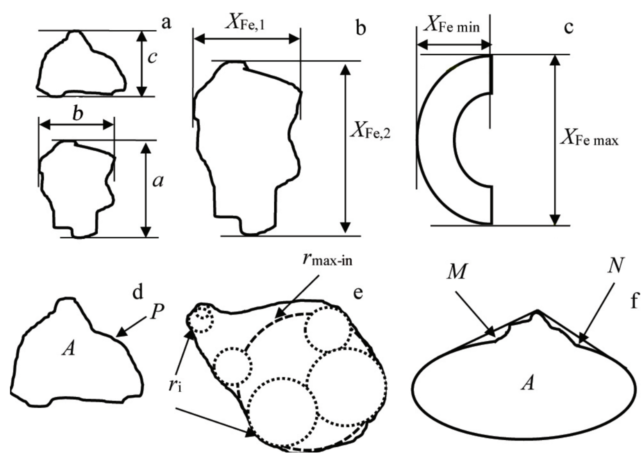

Fig.1 Schematic diagram of eight shape parameters: (a) Krumbein intercept sphericity, (b) Krumbein sphericity, (c) aspect ratio, (d) Compct & C & SPHT, (e) Krumbein roundness, and (f) convexity & solidity

$$\text{Solid} = \frac{A}{A_{\text{conv}}} \tag{9}$$

3 Results

3.1 Qualitative characterization (FE-SEM)

SEM images of three kinds of superalloy powders are shown in Fig.2. It can be seen that the 1# powder in different particle size ranges have high sphericity. The satellite particles can rarely be observed. Likewise, the capped particles are far beyond observation. The powder surface is smooth. Compared with the 1# powder, the 2# powder in the corresponding particle size range presents a more irregular shape, in terms of large amounts of satellite particles and low fractions of spherical particles. Meanwhile, the powder surface is not as smooth as 1# powder. The capped particles can be observed, but not many. Compared with the 1# and 2# powders, the 3# powder in the corresponding particle size range has the most irregular shape, that is, the largest amount of satellite particles and the lowest degree of sphericity. The powder surface is rougher than that of 1# and 2#. A large number of capped and agglomerated particles are observed.

Therefore, the comparisons among the three superalloy powders show that the shape of 1#, 2#, and 3# gradually deteriorate. Simultaneously, the sphericity decreases in succession. The powder surface becomes rougher. The quantity of capped particles increases progressively. Thus, the three samples used in this study cover a significant range of particle shape.

3.2 Quantitative characterization (DIA)

In order to quantitatively characterize the difference of particle shape, the dynamic image analysis (DIA) technique is applied. The corresponding results are shown in Fig. 3. The cumulative distribution curves of eight shape parameters almost demonstrate the similar increasing trend. However, the distinction degree is extremely different.

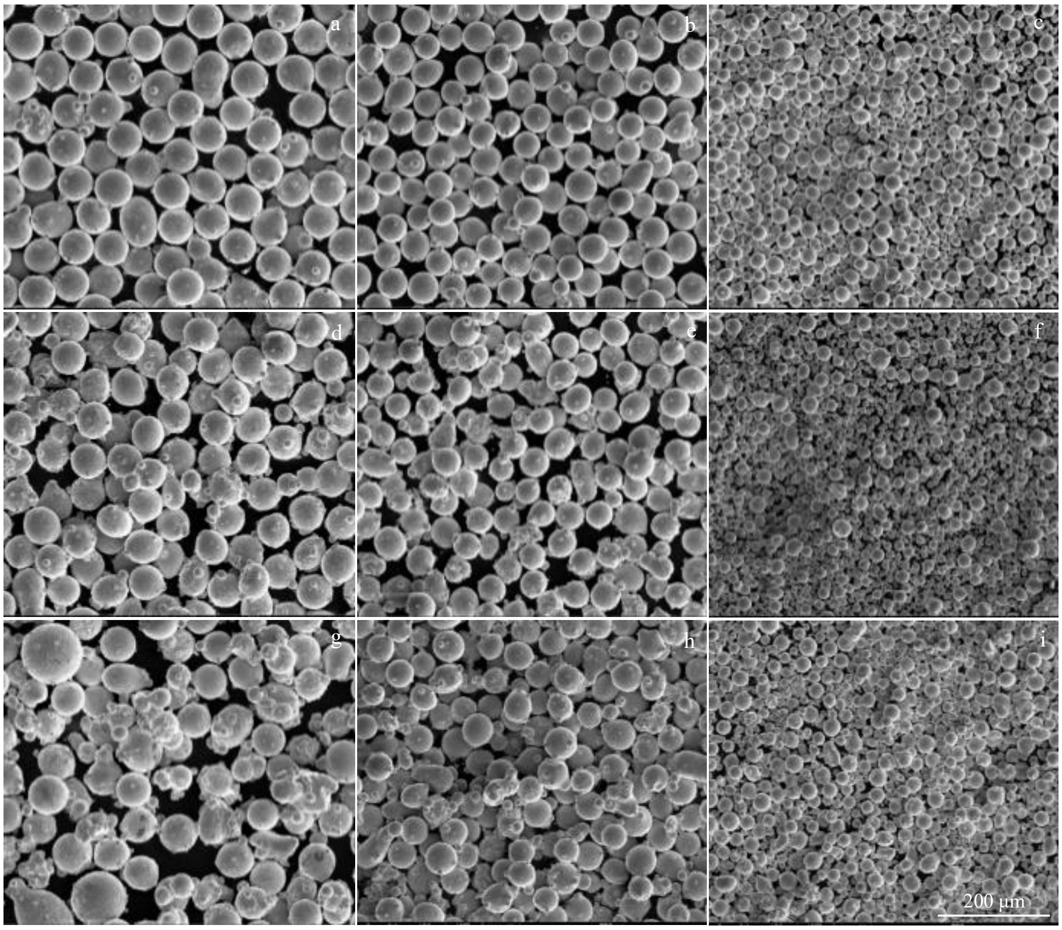

Fig.2 SEM images of three kinds of superalloy powders; (a) 1#, 53~60 μm, (b) 1#, 38~45 μm, (c) 1#, ＜25 μm; (d) 2#, 53~60 μm, (e) 2#, 38~45 μm, (f) 2#, ＜25 μm; (g) 3#, 53~60 μm, (h) 3#, 38~45 μm, (i) 3#, ＜25 μm

As for SPHT_K, its cumulative distribution curve of 1#, 2# and 3# changes considerably, indicating that the parameter has high distinction degree (Fig. 3a). The SPHT_K is a shape parameter which evaluates the variation of particle sphericity by the value of min($X_{Fe,1}/X_{Fe,2}$). The obvious difference of distinction degree represents the significant difference of sphericity among the three samples. Compared with SPHT_K, the AR and Compct change moderately, indicating that the AR and Compct have the relatively low distinction degree. The AR and Compct demonstrate similar distinction degree (Fig.3b and 3c).

Though the SPHT_K is a very accurate shape parameter to characterize the particle sphericity, there is an inadequacy that the parameter cannot distinguish the particle whose SPHT_K value is close to 1. However, the distinction degree of AR and Compct is much higher than that of the SPHT_K in the corresponding interval. Thus, the SPHT_K, AR and Compct should be combined together to characterize the particle shape variation in the full range. From the overall form aspect, these three shape parameters are recognized as the suitable shape parameters to characterize the variation of particle shape.

The distinction degree of RDNS_C cumulative distribution curve of 1# and 2# is very significant, but the distinction degree of 2# and 3# seems very small (Fig. 3d). These results indicate that the sharpness of particle corners is markedly different between 1# and 2#. However, the difference of 2# and 3# is much slighter than the variation of 1# and 2#. It represents that the sharpness of 2# and 3# contour is not much different. It is consistent with the results shown in Fig.2.

Compared with the RDNS_C, the four shape parameters C, SPHT, Conv_A and Solid have relatively low distinction degree, because these four shape parameters change slightly with the variation of particle shape. As shown in Fig. 3e~3g, the distinction of C, SPHT and Conv_A cumulative distribution curve of 1#, 2# and 3# is very slight, which cannot accurately characterize the difference of particle shape. Thus, the three shape parameters are excluded. From the more detailed aspect, the RDNS_C may be a proper parameter.

Even though the RDNS_C have a relatively low distinction degree between the 2# and 3#, the variation of 1# and 2# can be distinguished obviously. Thus, from the roundness aspect, the RDNS_C can be taken into account as the proper shape parameter to evaluate the difference of the smooth of particle outline. Furthermore, the four shape parameters C, SPHT, Conv_A and Solid change inconsiderably with the variation of particle shape. Therefore, they are not suitable to be selected

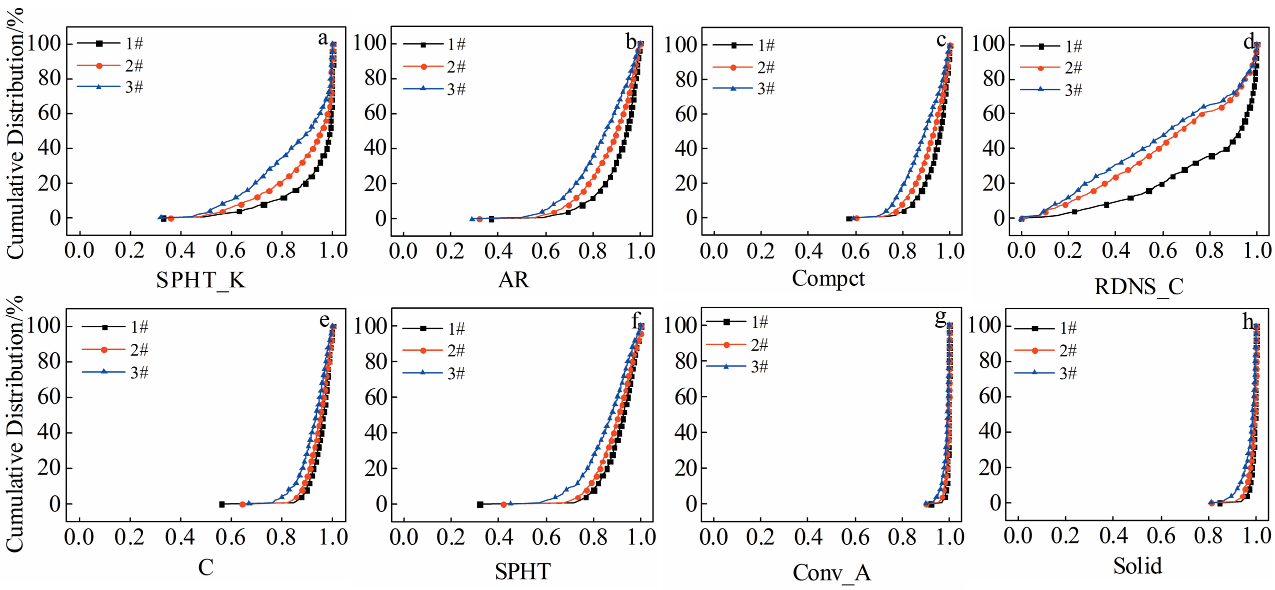

Fig.3 Eight shape parameters of three kinds of superalloy powders: (a) SPHT_K, (b) AR, (c) Compct, (d) RDNS_C, (e) C, (f) SPHT, (g) Conv_A, and (h) Solid

as the shape parameters to characterize the particle shape. In summary, the SPHT_K, AR, Compct and RDNS_C are selected to be the suitable shape parameters to characterize the particle shape.

The mean values of eight shape parameters of superalloy powders are shown in Table 1. The change trends of mean values of the eight parameters (Table 1) are consistent with the results in Fig.2 and Fig.3. The shape of 1#, 2# and 3# becomes worse in turn; for example, the number of satellite particles increases significantly. The mean values of the eight shape parameters decrease gradually; for example, the SPHT_K decreases from 0.936 to 0.894, and then drops to 0.847. The cumulative distribution curve of 1#, 2# and 3# move towards left in turn.

4 Discussion

4.1 Distinction degree of shape parameters

Quantitative characterization of particle shape is one of the most challenging issues in powder technology[38]. The shape of a three-dimensional object can be described by numerous shape parameters. For an in-depth understanding of the shape parameters, it is important to establish definitions regarding the basic characteristics of a three-dimensional object.

Barrett[39] recognized three independent particle shape parameters in scales (Fig. 4), including form, roundness, and surface texture. Form, the first order property, reflects variation in the proportions of the particle. It is a major characteristic which reflects the geometrical proportions of a particle. The property is usually calculated by dimensionless ratios. Roundness, the second order property superimposed on form, reflects the radius of curvature at the particle corners. As the third order effect, surface texture is taken for defining local roughness features at corners and edges between corners. It refers to finer irregularities superimposed on roundness and form[40-43].

Through the above definitions regarding the basic characteristics, the eight shape parameters can be classified into the following two categories: SPHT_K, AR and Compct belong to the macroshape descriptors which are related to overall form. RDNS_C, C, SPHT, Conv_A and Solid belong to the mesoshape descriptors which evaluate the sharpness of corners of particle.

As for the macroshape descriptors, the SPHT_K can considerably characterize the difference of particle shape. The reason may be that the SPHT_K is a macroshape descriptor which can present the overall shape of particle in a holistic approach. Thus, the distinction degree of SPHT_K seems the highest in the macroshape descriptors, characterizing the primary variation of particle shape.

As for the mesoshape descriptors, for example, the RDNS_C, C, SPHT, Conv_A and Solid, the difference of 2# and 3# is not very significant. However, the distinction degree of RDNS_C between 1# and 2# is pronounced enough to distinguish the difference of two samples. From the definition

Table 1 Mean values of shape parameters of superalloy powders

Sample	SPHT_K	AR	Compct	RDNS_C	C	SPHT	Conv_A	Solid
1#	0.936	0.909	0.937	0.800	0.953	0.910	0.995	0.988
2#	0.894	0.871	0.913	0.643	0.943	0.891	0.992	0.983
3#	0.847	0.833	0.883	0.604	0.927	0.855	0.986	0.972

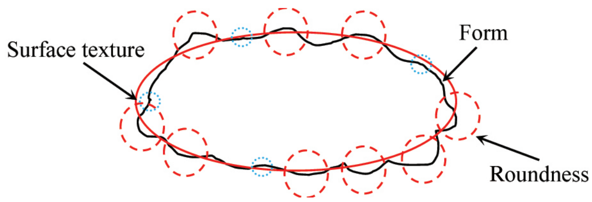

Fig.4 Particle shape parameters at different scales

of this parameter, it is clear that the RDNS_C is a parameter which is used to evaluate the sharpness of particle corners. Therefore, the great variation of RDNS_C between 1# and 2# indicates the obvious change of ratio between particle corner radius and the maximum inscribed circle radius, for example, the variation of satellite particles. Thus, the RDNS_C can be a suitable shape parameter to characterize the slight difference of particle shape.

4.2 Distribution of shape parameters

Xiu et al[44] indicated that the box plot can be used to characterize the distribution of shape parameters. In order to analyze the distinction degree and distribution of each shape parameter, the box plots for particle shape parameters comparison of samples are shown in Fig 5. The result in Fig.5 is consistent with Fig.3. Though the variation between 2# and 3# seems not very apparent, the RDNS_C has the high distinction degree and a wide distribution between the 1# and 2# particle shape. Thus, the RDNS_C can distinguish the difference of the three superalloy powders. The SPHT_K, AR and Compct remains at the middle change range. However, the variation values of the three shape parameters are very identifiable to characterize the particle shape effectively. Other shape parameters change moderately, including the C, SPHT, Conv_A and Solid. Therefore, they will not be selected as the proper shape parameters to characterize the variation of particle shape.

From the above results and discussion, it can be seen that the changing pattern of qualitative characterization of particle shape is consistent with the quantitative characterization. The SPHT_K, AR, Compct and RDNS_C which have a high distinction degree, can be the appropriate shape parameters to characterize the overall and detailed variation of particle shape.

4.3 Significance of four shape parameters

Some researchers have shown that particle shape, size, and other mesoscopic factors influence the mechanical property of granular materials more strongly than other factors[45,46]. Among these factors, the particle shape may be one of the most important parameters that affect the mechanical property of particle materials. In the previous studies, the particle shape is qualitatively characterized by the FE-SEM, which cannot evaluate the particle shape through quantification. In this study, we extracted four important shape parameters (the SPHT_K, AR, Compct, RDNS_C) to quantitatively characterize the variations of particle shape. This shape parameter combination will substantially promote the improvement of the mechanical properties of powder metallurgy and 3D printing products, and thus accelerate the application and development of additive manufacturing and advanced powder metallurgy technology. The implication of accurate characterization of the particle shape by the four shape parameters is widely recognized, and discussed as follows.

As a macroscopic characterization parameter, the Krumbein sphericity (SPHT_K) characterizes the overall shape of particle from a global perspective. This shape parameter basically evaluates the macrostate. The shape parameter aspect ratio (AR) is selected to characterize the elongation of the particle in the projection plane. The compactness (Compct) evaluates the degree a particle similar to a circle, considering the overall form of the particle.

As for the mesoscopic shape parameter, the Krumbein roundness (RDNS_C) can be used to evaluate the sharpness of the particle contour. This parameter is particularly sensitive to changes in the amount of satellite powder.

From all the two aspects to characterize the particle shape, the particle shape of superalloy powder used in powder metallurgy and 3D printing will be accurately characterized and described. Thus, the atomization processing parameters

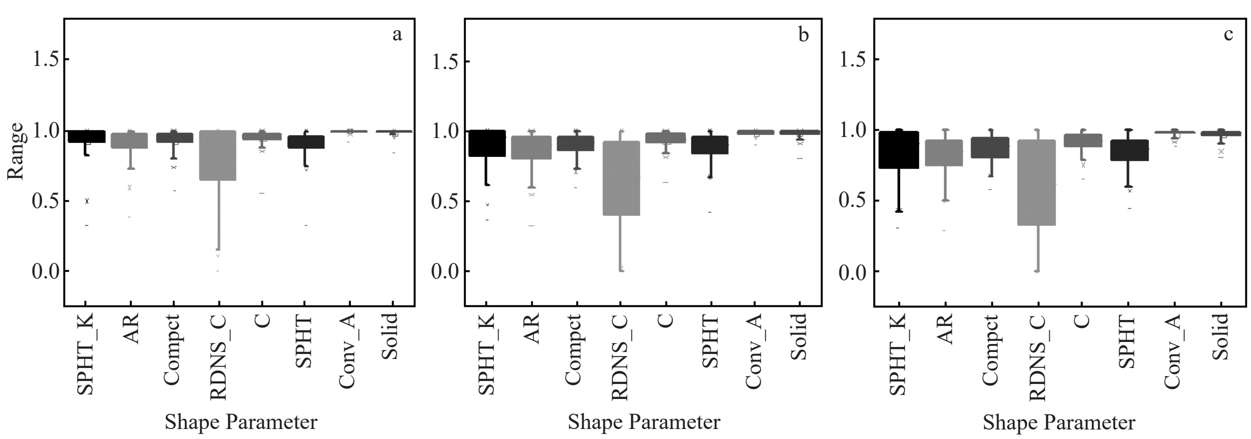

Fig.5 Box plots of shape parameters for different samples: (a) 1#, (b) 2#, and (c) 3#

will be adjusted in order to control the particle shape. In addition, the mechanical property of the alloys fabricated by the optimized superalloy powder will be improved significantly. Meanwhile, the shape parameter combination provides insights into the choice of specialized requirement for particle shape or the related fields.

5 Conclusions

1) The SPHT_K, AR, Compct and RDNS_C have high distinction degree and can be used as the shape parameter combination to evaluate the variation of particle shape.

2) The shape parameter combination can quantitatively characterize and control the particle shape.

3) This combination will promote the application and development of additive manufacturing and advanced powder metallurgy technology.

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采用动态图像分析技术对高温合金粉末形状定量表征

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摘　要：采用场发射扫描电子显微镜（FE-SEM）和动态图像分析（DIA）技术，选用8个形状参数对高温合金粉末形状进行了定性及定量表征。结果表明，Krumbein球形度（SPHT_K），长宽比（AR），紧凑度（Compct）和Krumbein圆润度（RDNS_C）对高温合金粉末形状具有显著的区分度，可以作为粉末形状表征的关键参数使用。此外，发现圆度（C）、球形度（SPHT）、SPHT_K三者之间的区分度依次增加，尤其是SPHT_K的区分度最高。通过对比8个形状表征参数的区分度差异，发现C、SPHT，凹凸度（Conv_A）和坚固性（Solid）的区分度并不显著。最终，将SPHT_K，AR，Compct和RDNS_C 4个参数组合用于定量表征高温合金粉末颗粒形状变化，为雾化参数的有效调整提供粉末形状的定量表征依据，促进增材制造和先进粉末冶金技术的研发。

关键词：高温合金粉末；颗粒形状；定量表征；动态图像分析

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