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Fractal phenomena in powder injection molding process①

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Abstract: The complicated characteristics of the powder were studied by fractal theory. It is illustrated that powder shape, binder structure, feedstock and mold-filling flow in powder injection molding process possess obvious fractal characteristics. Based on the result of SEM, the fractal dimensions of the projected boundary of carbonylic iron and carbonylic nickel particles were determined to be 1.074 ±0.006 and 1.230 ±0.005 respectively by box counting measurement. The results show that the fractal dimension of the projected boundary of carbonylic iron particles is close to smooth curve of one-dimension, while the fractal dimension of the projected boundary of carbonylic nickel particle is close to that of trisection Koch curve, indicating that the shape characteristics of carbonylic nickel particles can be described and analyzed by the characteristics of trisection Koch curve. It is also proposed that the fractal theory can be applied in the research of powder injection molding in four aspects.

Key words: powder injection molding; fractal theory; chaos; Navier-Stokes equation
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1 INTRODUCTION

Generally, The filling process of powder injection molding(PIM) is analyzed by rheological theory. Rheological model is made up of basic conservation equations(mass, momentum, energy conservation equations) that must be satisfied by melt feedstock in PIM, constitutive equation that is used to describe the processing parameters of materials, and corresponding restrictive conditions. It is a group of partial differential equations and is also a nonlinear dynamics system of many influential factors. The variation of material thermophyical and rheological properties caused by the fluctuation of process parameters may result in multivaluedness of stress—strain curve, that is, the fluctuation of process parameters result in the bifurcation. In PIM, mutations(defects arise) of flow state caused by the small variation of process parameters may correlate to the chaos of nonlinear dynamics system[1].

2 FRACTAL THEORY AND FRACTAL CHARACTERISTICS OF POWDER

Chaos is a major class of order phenomena without periodic. The important characteristics of chaos motion are sensitively dependent on the variation of initial values, that is, in nonlinear dynamics system, tiny variation of initial values will lead to totally different type motion. Fractal theory is one of the geometry languages used to describe chaos kinetics. Fractal theory and chaos theory are closely correlative. Fractal theory reveals the unity of order and disorder and that of certainty and randomness in nonlinear system. Fractal theory was first proposed in 1970s. But through more than 20 years development, it has been an important new branch of studying and extensively applied in almost every field of the natural science and social science. Nowadays, fractal theory has been one of the front research subjects in many sciences. Mandelbrot[2] proposed fractal geometry to describe and investigate these extremely irregular and fragmental geometry objects in nature. The research objects of fractal theory have two common characteristics. One is not having characteristic length; the other is having self-similarity between the part and the whole. Take natural shoreline and Koch curve for example, no

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matter how to amplify it, the part is as intricate as before, and the intricate shapes contain organizational structure. The mathematics essence of fractal theory lies in that it alters mearsure view of observing and researching objects, such as the replacement of Euclidean measure with Hausdorff measure. Because of this change of measure view, we can reveal the self-similarity or self-affine rules that exist in complex phenomena and be more powerful to learn and deal with complex natural phenomena.

There are many refined and complex fractal phenomena in PIM[3]. Through observing PIM processing and analyzing rules of defect occurrence during green part forming, QU et al[1] pointed out that phenomena such as defect occurrence of PIM may be correlative to chaos phenomena in nonlinear dynamics system, and proposed that fractal theory and chaos can be applied to the research on PIM. Micrographs of projected boundary of powder particle, surface, pores of apparent and tap powders can be obtained by scanning electron micrograph(SEM) or optical microscope(OM). Fig. 1 shows the agglomeration, size, and shape of some typical powders used in PIM at low and high magnifications, showing that agglomeration, size, shape, surface and spatial structure of powders are complex. The particle surface is tortuous and fragmental with the presence of tiny isolated islands. Actually there is a lot of information of powder particle in these phenomena. In the past, in both numerical computation and experimental research, these phenomena are described or assumed as continuous, smooth curves(plane), which in fact neglects much important detail information. The detail information may be exactly the important factors of influencing the process of PIM. The research which fractal theory and chaos are applied in PIM is helpful to describe and analyze the important and complicated detail information.

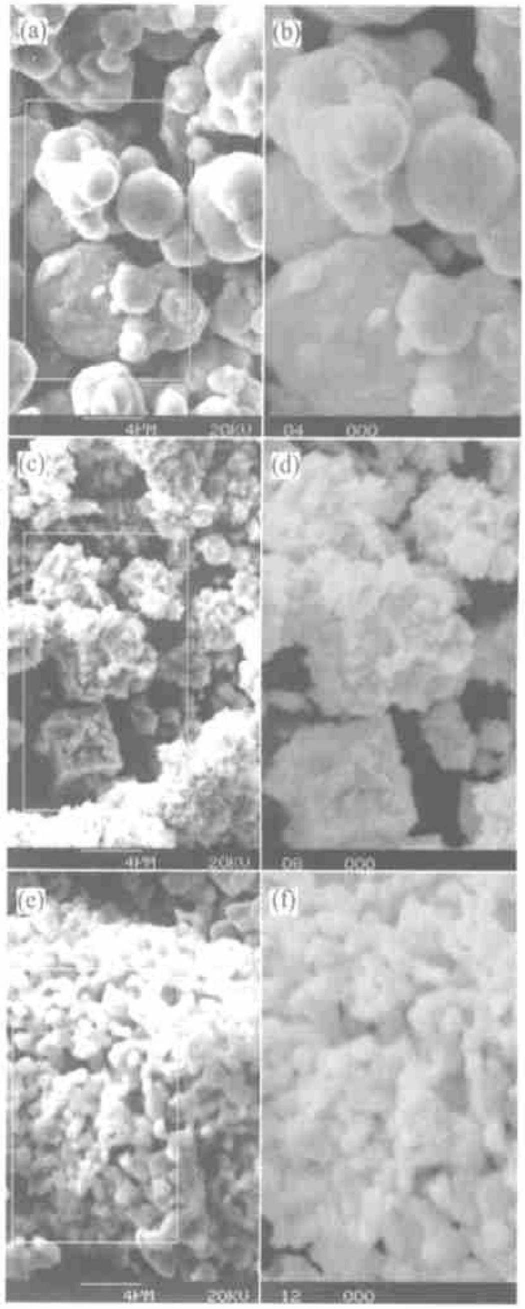

Fig. 1 SEM photographs of powders used in PIM
(a) —Carbonylic iron at low magnification;
(b) —Carbonylic iron at high magnification;
(c) —Carbonylic nickel at low magnification;
(d) —Carbonylic nickel at high magnification;
(e) —Reduced cobalt at low magnification;
(f) —Reduced cobalt at high magnification

3 FRACTAL PHENOMENA IN PIM PROCESS

There are many tiny and complicated fractal phenomena in PIM, such as shape, surface, cross section of powder particle, and spatial structure formed by the pores of apparent and tap powder etc.

3.1 Fractal phenomena of powder particle

A particle is defined as the smallest unit of a powder that can not be subdivided by simple mechanical means. In PIM, the particles generally are below 20 μm in size. The powders used in PIM have fractional packing density between 0.3 and 0.8 of theoretical density, with a typical level near 0.6. The optimal solids content for molding depends on the powder characteristics, particle size distribution, particle shape, interparticle friction and agglomeration[4-6]. Particle size can be measured by determining the dimensions of a particle. The size depends on the measurement technique, specific parameter being measured, and particle shape. It can be achieved by several techniques which usually do not give equivalent determinations, due to differences in the measured parameters. One geometric parameter is used and the assumption of a spherical particle shape is made durring measuring process. The analysis parameters are surface area, projected area, maximum dimension, minimum cross sectional area or volume. This analysis is based on Euclidean measure.

As the shape becomes less regular, the numbers of possible size parameters increase and the determination of particle size becomes more difficult. Thus, it is common to find disagreement between the particle size distributions obtained by different techniques and instruments, and the different techniques or instruments are applicable to the different particle sizes. Almost all the techniques and instruments based on Euclidean measure are not applicable to the very small particles whose Euclidean yardstick is below 0.1 μm. This demonstrates that the Euclidean view can not be used to reasonably describe the characteristics of particles which have the "infinitely refined" fractal structure and yardstick size less than 0.1 μm. This is just like that Koch curve or shoreline can not be described reasonably by Euclidean measure, because the self-similarity dimension of Koch curve is not an integer, but a fraction between 1 and 2. For example, the self-similarity dimension of trisection Koch curve $D = \ln 4/\ln 3 \approx 1.262$. It can be inferred that the fractal dimensions of particle surface area, projected area, and minimum cross sectional area are not integer. The boundary curve of projected area of particle is similar to that of Koch curve. The surface powder particle is rather intricate, and its fractal dimension may be between 2 and 3. German[4] pointed out that very small particles, whose Euclidean yardstick is below 0.1 μm, could be characterized by analysis of their Brownain motion in a fluid. The nonuniform molecular collisions on the particle surface cause the particles to move randomly in inverse proportion to the particle mass. The measurement of the Brownian motion provides particle size information through the particle velocity detected by a Doppler shift in scattered laser-light. In fact, the track of Brown motion has typical fractal property, only that its fractal dimension is 2, still an integer[7]. These phenomena and facts demonstrate that PIM powder particles have the outstanding fractal characteristics. To describe particle size and shape that embody powder particle property sufficiently, it is necessary to apply the views and concepts of fractal geometry.

3.2 Fractal phenomena of binder and PIM feedstock mixture

During PIM process, the binder is a temporary vehicle for homogeneously packing the powder into the desired shape and then holding the particles in that shape until the beginning of sintering. Although the binder does not dictate the final composition, it has a major influence on the success of processing. Binder compositions and debinding techniques are the main differences between various PIM processes[4, 8, 9].

The binder is mixed with the powder to form a uniform feedstock for molding. It has an influence on particle packing, agglomeration, mixing, rheology, molding, debinding, dimensional accuracy, defects, and final chemical property of PIM compacts. There are many types of binders, and most of which are polymers. Polymers are long-chain molecules with carbon backbones and various side groups and branches. Thermoplastic and thermosetting compounds are the two general forms of polymers. The longer-chain polymers have the amorphous characteristics, that is, fractal characteristics. Beside thermoplastic component, binder contains additives used to lubricate and control viscosity, humidification and debinding. Iacocca et al[10] used deterministic chaos theory to evaluate the function of lubricant in filling process. This demonstrates that binder has fractal characteristics. The properties of feedstock vary dramatically with the increase of powder content in feedstock, such as the increase of elastic modulus, strength and viscosity, the decrease of thermal expansion coefficient and ductility. It is difficult to precisely describe the influence of particle characteristics, temperature and binder characteristics applied on powder-binder system. The results obtained at present are mostly empirical formulas. Because the particle shape and binder have fractal characteristics, and PIM feedstock has corresponding fractal characteristics, too. These fractal characteristics will be investigated and analyzed with fractal viewpoint and concepts.

3.3 Fractal phenomena in PIM filling process

The process of PIM filling is a viscoelastic, unsteady, non-isothermal complex physical process. Addition to the complicated flow geometry shape of the die cavities, it is very hard to describe the process of feedstock flow precisely. At present, common mathematical models used to describe the process of PIM filling are mainly based on the following assumptions[3].

1) Powder and binder are mixed well without any gas hole, and the mixture never separates du-ring the flow. The feedstock melt fluid flow is considered as even continuous medium non-Newtonian fluid flow, and the effects of heat expansion and the latent heat can be neglected.

2) Heat conduction plays an important role in cavity wall, and the convective transmit heat in cavity thickness direction is neglected. While the convective transmit heat plays the greatest role in cavity, and heat conduction in the stream direction in cavity is neglected.

3) In the cavity, viscosity is considered only, and inertial force, elasticity and gravity are neglect-

ed. The pressure is assumed to be constant in the thickness direction.

According to these assumptions, a group of Navier-Stokes equations (N-S equations) based on continuous medium model and used to describe PIM process are obtained. Models based on other assumptions, such as granular model, two-phase flow model, are more complex N-S equations. Except some classical differential equations, it is difficult to certify whether the solution of equations leads to chaos phenomenon from differential equations. Many chaos phenomena of differential equation solutions are discovered and demonstrated by numerical solutions. For the flowing of PIM filling process, many numerical solutions based on continuous medium indicate that the flowing is dramatically influenced by processing parameters, and its pressure distribution, temperature distribution etc have fractal characteristics. N-S equations are correlative to fractal and chaotic phenomena, and can be explained by the classical Lorenz equation. Lorenz[11] studied the atmospheric turbulence and obtained a group of ordinary differential equations called classical Lorenz equation by simplifying N-S equations vastly. Chaotic behavior and Lorenz attractor have been discovered through solving the numerical solution of the equation. The attractor is a fractal set, and its fractal dimension is 2.06[12]. Scheffer studied the status of solutions of N-S equations in three-dimensional space, and found that the oddness of equation solution (if exist) must be in the set whose dimension is less than 3[13]. It is illustrated by experiment study and production practice for years that the same defects of PIM products have self-similarity and the occurrence of the defects of PIM products is correlative to the small variation of processing parameters. These facts sufficiently demonstrate that PIM filling have the outstanding fractal characteristics and chaotic phenomena.

Although N-S equations are correlative to fractal, there is no method that can be used to deduce the fractal dimension from these basic equations directly. Especially in PIM flowing process, the occurrence of the chaotic phenomena has relation to the processing parameters, the characteristics of feedstock etc. The fractal characteristics and chaotic phenomena of PIM filling process can be studied by numerical computation and computer simulation.

4 FRACTAL DIMENSION USED TO RESEARCH FRACTAL CHARACTERISTICS OF PIM AND EXPERIMENTAL MEASURE METHODS

Fractal dimension is one of the important parameters of fractal structure. Theoretical researchers devote to analyzing all kinds of fractal structure and their forming processes, computing fractal dimensions which express their characteristics; while the experimental researchers measure the fractal structure and fractal dimension of its process by experimental measure methods, and find out the relationship between fractal dimension and material property or even further discuss the physical reasons for the formation of fractal structure. Fractal dimension is also a fundamental quantity in fractal theory. For the reason that it is difficult to define the fractal dimension applied to all aspects, there are many methods for defining fractal dimension in documents and books, such as relevant dimension, informative dimension, self-similar dimension, Hausdorff dimension, counting-box dimension and Kolomogrov capacitive dimension etc. Different definition methods applied to different studying objects, and the fractal concept and its dimension are kept on developing.

4.1 Hausdorff dimension

Assume $D > 0$, and cover the set S with balls whose diameter is ε and quantity is $N(\varepsilon)$ and so the D-dimension measure M_D of set S is defined as

$$M_D = \lim_{\varepsilon \to 0} \gamma(D) N(\varepsilon) \varepsilon^D,$$

where $\gamma(D)$ is geometrical factor. For straight line, square and cube, $\gamma(D) = 1$. For disk and sphere, $\gamma(D) = \pi/4$ and $\pi/6$, respectively; D is called Hausdorff dimension or Hausdorff-Besicovitch dimension of set S. For self-similarity set, Hausdorff-Besicovitch dimension equals self-similarity dimension. Here the "ball" and "diameter" are abstract concepts, which may represent straight line, square, cube, disk and sphere etc.

4.2 Counting-box dimension

Assume $S \subset \mathbf{R}^n$. In the Euclidean distance, cover the set S with little boxes whose side length equals $\frac{1}{2^n}$, and let $N_n(S)$ be the minimum number of box covering S, then

$$D = \lim_{n \to \infty} \frac{\ln N_n(S)}{\ln 2^n},$$

where D is the counting-box dimension of set S.

4.3 Experimental measure methods of fractal dimension in PIM

There are many practical measure methods of fractal dimension, such as perpendicular section, island, surface area direct measurement, power spectrum, secondary electron scatter etc[7]. Different methods are used depending on different research objects. One simple and practical method is the rough visual method which gets the value of the dimension by changing the degree of the rough visibility. Its important thought is based on that the details of fractal set is reflected by choosing the yardstick, and the smaller the yardstick is, the more the details are reflected. Box-counting method is one of the rough visual methods.

Divide the fractal graphics by quadrate mesh with ε in mesh width, as illustrated in Fig. 2. This step is called space quantization. Then count out the quadrate mesh number $N(\varepsilon)$ contained in the research scope. In the images processing, this is to calculate the number of all meshes containing image element of fractal graphics, and the fractal dimension D can be calculated by $N(\varepsilon) \propto \varepsilon^{-D}$.

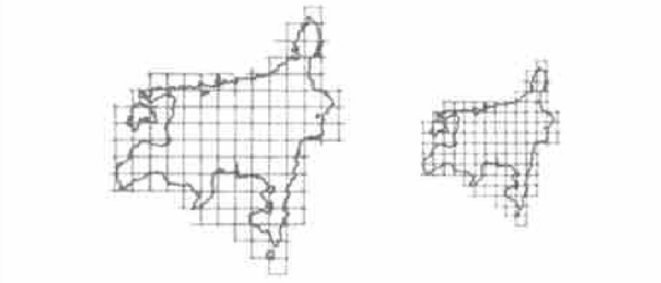

Fig. 2 Similar islands measured by relative yardstick measure

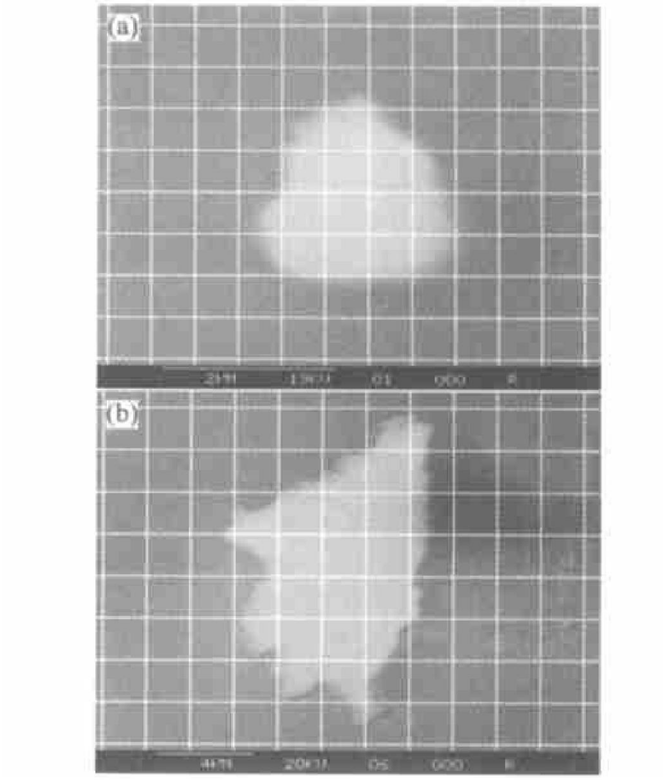

Fig. 3 Projections of two powder particles
(a) —Carbonylic iron; (b) —Carbonylic nickel

5 FRACTAL DIMENSIONS OF BOUNDARY CURVE OF CARBONYLIC IRON AND CARBONYLIC NICKEL POWDER PARTICLES

SEM is one of the best tools available for observing the discrete characteristics of a particle. Fig. 1 shows the scanning electron micrographs of carbonylic iron and carbonylic nickle powders used in PIM processing at low and high magnifications, showing the agglomeration, size, shape and the complexity of surface and spatial structure of powder particle. From the SEM images of carbonylic iron and carbonylic nickel powders, the approximate qualitative description of particle characteristics of the two powders can be obtained. Also, the aspect ratio and the sphericity index can be used to simply quantitativly describe them. But obviously it is difficult to describe the difference of particle shape, complication of agglomeration, surface and spatial structure complexity of the two powders in detail, resulting in difficulty in exactly controlling the processing of PIM.

Particle shape relates to the characteristics of the material and powder fabrication approach etc. Carbonylic iron and carbonylic nickel powders are separated by ultrasonic wave, and through their SEM images, it is found that the projected boundary of the same kind of powder particles has the similarity, and it has very complicated fractal structure. Fig. 3 shows the typical projections of carbo-nylic iron powders and carbonylic nickel powders by SEM. The fractal dimensions of the projected boundary of carbonylic iron and carbonylic nickel powders are calculated by the counting-box method.

5.1 Fractal dimension of projected boundary of carbonylic iron particle

The projection of the carbonylic iron particle is parted by quadrate mesh with ε in mesh width, as shown in Fig. 4. The length of the square in Fig. 4 (a) is considered as an unit length (the absolute length is $\varepsilon = 100/36$ μm). With the decrease of relative yardstick ε, count out the quadrate mesh number $N(\varepsilon)$ contained in the projection of carbo-nylic iron particle. The relation of ε—$N(\varepsilon)$ is listed in Table 1. Therefore the double logarithmic relation of ε—$N(\varepsilon)$ can be obtained (shown in Table 2).

Make curve fitting for data about the double logarithmic relation of ε—$N(\varepsilon)$ of a great deal of carbonylic iron particles with straight lines, the fractal dimensions of the boundary of carbonylic iron particles are calculated to be $D = 1.074 \pm 0.006$, which is close to 1.1. The results show

Table 1 ε—$N(\varepsilon)$ relation of carbonylic iron powder

ε	1	34/80	34/200	34/1 000	34/10 000
$N(\varepsilon)$	1	6	16	91	1 075

Table 2 $\ln\varepsilon$—$\ln N(\varepsilon)$ relation of carbonylic iron powder

$\ln\varepsilon$	0	−0.856	−1.772	−3.381	−5.684
$\ln N(\varepsilon)$	0	1.792	2.773	4.511	6.980

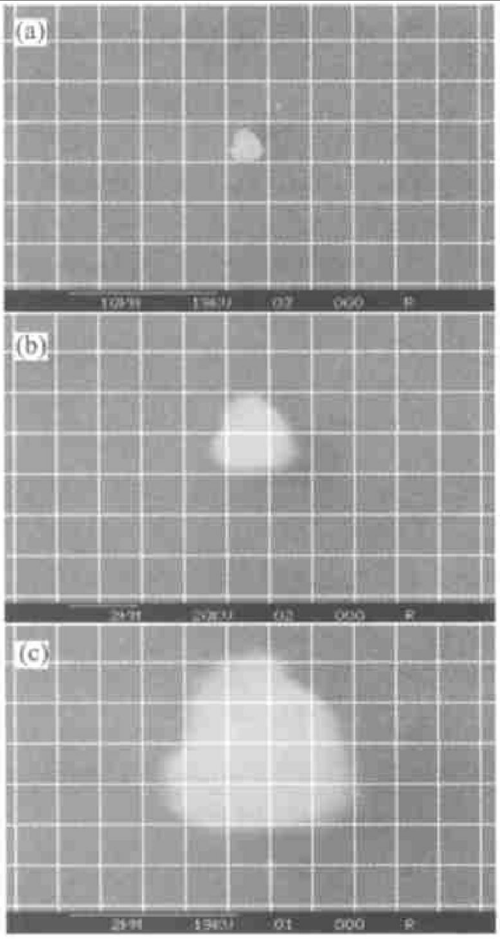

Fig. 4 Projections of carbonylic iron particle by quadrate mesh
(a) —With ε= 100/36 μm in mesh width;
(b) —With ε= 85/72 μm in mesh width;
(c) —With ε= 17/36 μm in mesh width

that the boundary of carbonylic iron particle is as near as smooth curve of one-dimension, but it still has obvious fractal structure.

5.2 Fractal dimension of projected boundary of carbonylic nickel particle

The projection of the carbonylic nickel particle is parted by quadrate mesh with ε in mesh width, as shown in Fig. 5. Use the same way above, the absolute length 12 μm or so is defined as the first relative yardstick ε.

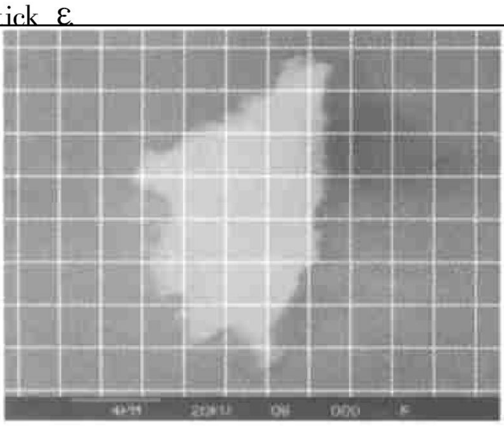

Fig. 5 Projection of carbonylic nickel particle by quadrate mesh with ε in mesh width

With the decrease of relative yardstick ε, count out the quadrate mesh number $N(\varepsilon)$ contained in the projection of carbonylic nickel particle. The relation of $\varepsilon - N(\varepsilon)$ is listed in Table 3. And the double logarithmic relation of $\varepsilon - N(\varepsilon)$ can be obtained (listed in Table 4).

Table 3 $\varepsilon - N(\varepsilon)$ relation of carbonylic nickel powder

ε	1	1/3	10/81	1/81
$N(\varepsilon)$	1	8	26	469

Table 4 $\ln\varepsilon - \ln N(\varepsilon)$ relation of carbonylic nickel powder

$\ln\varepsilon$	0	−1.099	−2.092	−4.395
$\ln N(\varepsilon)$	0	2.079	3.258	6.150

Make curve fitting for data about the double logarithmic relation of $\varepsilon - N(\varepsilon)$ of a great deal of carbonylic nickel particles with straight lines, the fractal dimensions of the boundary of carbonylic nickel particles are calculated to be $D = 1.230 \pm 0.005$, which is close to that of trisection Koch curve (1.262). The results show that the complexity degree of the boundary of carbonylic nickel particle is as near as that of trisection Koch curve. And from the SEM photographs of carbonylic nickel particles, it can be seen directly that the projections of carbonylic nickel particles are very close to the trisection Koch island in shape. Therefore the shape characteristics of carbonylic nickel particles can be described and analyzed by the characteristics of trisection Koch curve.

6 APPLICATION PROSPECT ON FRACTAL THEORY AND CHAOS IN PIM

The application of fractal theory and chaos in PIM can roughly be divided into four aspects: the first is to measure the fractal dimension of binder and particle shape by experimentation, to analyze the physical reason for the fractal structure forming of particle, and to describe the characteristics of particle shape more precisely; the second is to analyze the fractal characteristics of feedstock and its filling processing turbulence; the third is to approach the fractal chaos phenomenon of Navier-Stockes equations and the mechanism for the occurrence of defects of the product of PIM; and the fourth is to study the effects of the fractal structure of particle shape and binder on debinding and sintering. In conclusion, the fractal structure and chaos phenomena may arise in the whole process of PIM. The study of PIM using fractal and chaos theory provides the new view and method to describe the material characteristics and process parameters, to more precisely control the process of PIM, and to achieve the goal for high quality and precise production.

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