Researches on new technology of diamond micron powder

S.D.Zhang
Tianjin Research Center for Superhard Abrasives,1,yingshui Dao.Tianjin,300191,China
New York Academy of Sciences, 2 East 63rd St, NY, 10021, USA

Abstract

The new manufacture technique of diamond micron powder is introduced in the paper. It is given that the principles and process parameters on broken and shaped, chemical treatment, skill for size separating operation etc. The regent No.13 and the separation liquid No.14 has been designed and adopted. The product made by the method has been come up to the advanced level in the world.

keyword: diamond, micron powder, technology

1.Introduction

Diamond micron powder is made from sub-millimeter diamond crystal, which is treated by broken, shaped and a serials of physical and chemical methods, and became regular shaped particles according with a desirable size distribution. Now diamond micron powder is the most advanced and precision superhard abrasive in the world,which is available for many areas, such as machinery, electronics, metallurgy, architecture, national defense and so on. It always processes hard-brittle materials and high precision parts. It could be used by abrasive, grain, paste, polishing suspension and paper. The consumption per year of diamond micron powder has become one of the indexes that measures the economic strength and scientific and technological level of a country. The output of diamond micron powder has increased quickly along with the raped development of advanced science and technology of the whole world. According to a rough estimate, diamond micronpowder's output has increased by two orders in ten years.

2.Distribution Process of SDZ-Method

The process is as follows: Diamond raw material → melting alkali treatment → cleaning → acid treatment → cleaning → magnetic selection → broken and shaped → acid treatment → cleaning → ultrasonic treatment → size separation → prime test → meltingalkali treatment → cleaning → acid treatment → cleaning→wasting and dispersion → packing and storage.

3.The design for ball mill type QM-631 and selection of the best process parameters

In the classical theory[1],the main parameters of ball mill are as follows: when machining hard and brittle materials, Diameter:Length>3; rotational speed $n=0.7\sim0.75n_{cri}$(critical rotation speed); ball :materials=2.5;the volume of the materials packed is 10~20% of the ball mill; and the volume of the ball is 40~50% of the ball mill; it total charge ratio 0.5~0.7. Making the diamond micron powder by above theory, single grain shape is not good and the quantity of the needle and platelike has exceeded by 10%.

In order to settle the problem of grain shape, first we determined and calculated the critical energy which single diamond grain of the highest strength has been broken. It equals 0.25J. After many experiments, the main parameters of ball mill are as follows: D:L=1, $n=0.9n_{cri}$, ball: materials=5~8, the total charge ratio =0.2~0.3, filling up raw material 50~80 thousand carats per batch.

Used ball mill type QM-631 and the parameters above, the single grain shape of the diamond micron powder has come up to advanced world standards. According to China and American standards, the amount of the needle and the palletize is 3%+5% and 3%+10% (to count large gains only)of the total grains respectively. Using our new process, the needle and the platelike is not observed (Table 1) both large grain and basic grain and fine grain.

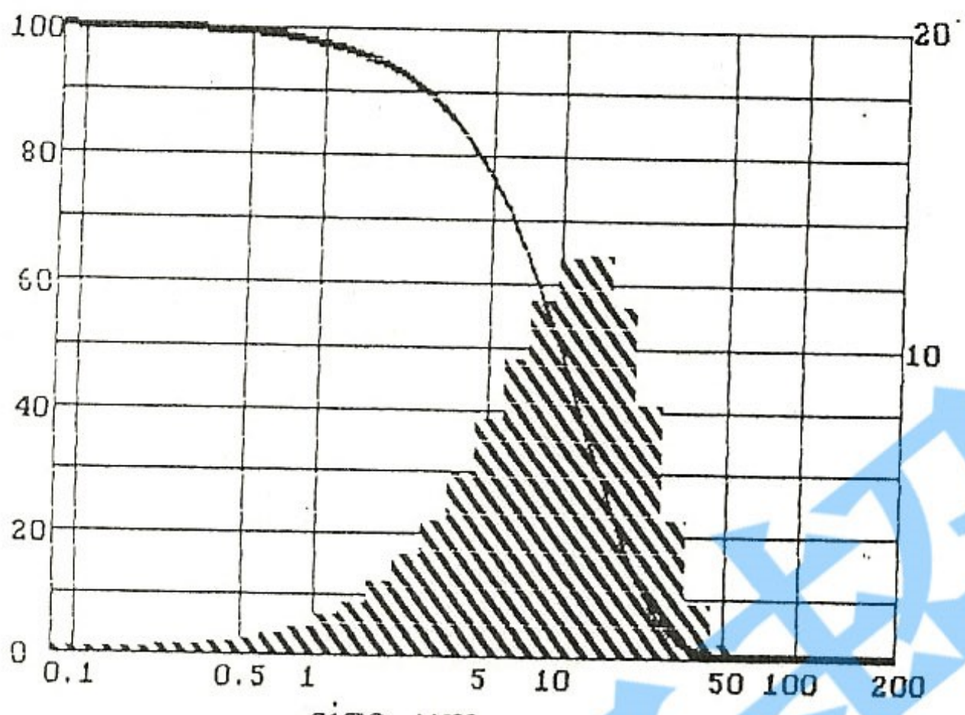
Table 1.
Grain shape of diamond micron powder ratio of needle and platelike

Symbol	ANSI B74.20-1981	GB6966.1-6966.5-86	SDZ Method
M0/0.75	3%+10%	3%+5%	0
M3.5/7	3%+10%	3%+5%	0
M53/75	3%+10%	exceeding size range	0

4. Using opposed jet mill

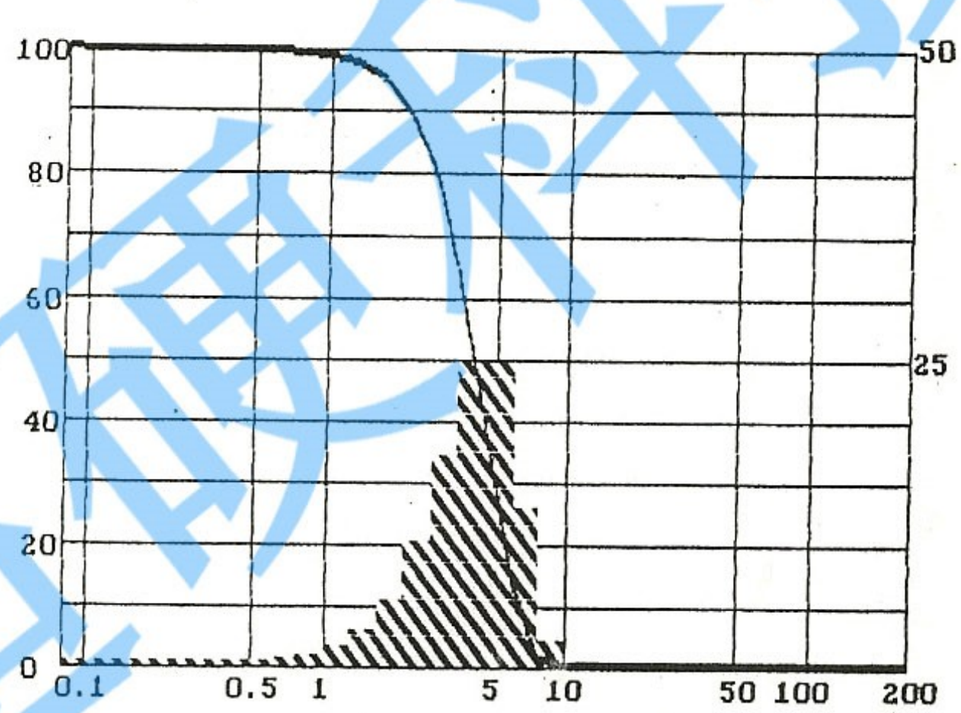
The technique that the materials have been broken by high press air current has been rapidly developed since
1960s. The opposed jet mill has many advantages: high product efficiency, continuous breaking, the different size has been prime classified simultaneously and so on. There is a classifying wheel in a opposed jet mill. According normal theory, if rotational speed of classifying wheel has been fixed, the size of the appointing material broken will be less than some numerical value.

In fact, feed speed of opposed jet mill has effect on the output size distinctly. Now we are breaking diamond by the type QLM-100 while classifying wheel speed is at 18000r/min. The experimental results are shown in the Fig .1. while feed speed of sample NQ1.0 kg/h and NQ 0.45 kg/h is 1.0 kg/h and 0.45kg/h respectively, their D_{50} are 9.29 μm and 4.01μm as well as D3 are 28.4μm and 7.05μm respectively. It is clear that influence of feed speed is much more than classifying wheels.



D_{95} 1.66, D_{90}2.65, D_{50}9.29, D_{10}21.2, $D_3$28.4, D_v10.8.

(a) feed speed 1.0kg/h



D_{95} 1.73, D_{90}2.18, D_{50}4.01, D_{10}6.17, $D_3$7.05, D_v4.09.

(b) feed speed 0.45kg/h

Fig.1. Output size distribution of different feed speed of opposite jet mill

Table 2.

Contrast test of treatment M0/1

Heating time. min	0	15	30	35	50	55	65	80	85
$HClO_4$ +K_2CrO_7 reagent NO.13	not reaction warm	not reaction light steam and foam	start light steam heavy steam	light steam light boiling	light boiling liquid has appeared red-yellow	light boiling the same as left, finish	boiling	liquid has appeared red	the same as left, finish

5. Special chemical treatment

Clearing the graphite out of the diamond micron powder, for example, it can be oxidation by perchloric acid. The formula is follows:

$$7C+4HClO_4=2Cl_2\uparrow+7CO_2\uparrow+2H_2O \quad (1)$$

Now, current process is to add $K_2Cr_2O_7$ and $HClO_4$. The liquid keeps dark green until C was oxidized completely. Finally, the liquid appears red color of Cr^{6+}. But the reaction speed is much slower. The reagent No.13 can quickly oxidate the graphite(Table.2). In brief, the time for acid treating was cut down about 1/3 and product cost decreased 40~50% using the reagent No.13.

There is no given standard of surface impurities in various countries, but its concentration is very significant for producing the sintered of diamond and diamond tools.

When the surface impurities is more, it is seriously affected to making diamond tools. For example, when making electroplating tools, it could be cause formed tumors.

The mainly non-metal impurities in diamond powder is pyrophyllite, which belongs to layer silicate mineral. It can be written as $Al_2O_3.4SiO_2.H_2O$. The reaction formula of melting alkali treatment is as follows:

$$Al_2O_3.SiO_2.H_2O+10NaOH = 4NaSiO_3+2NaAlO_2+6H_2O \quad (2)$$

For instance, silicon is comboned stability with carbon on diamond surface, C-Si bond energy is 301 kJ/mol. Only one time melting alkali treatment can basically clear away the pyrophyllite grain, but it can not completely get rid of the silicon which exists

on the surface of diamond particle as chemical bond.

Tow times melting alkali treatment has cleared nonmetal out of diamond micron powder. After the special chemical treatment, total amount of the impurities is <1% (Table.3) and surface impurities decrease by one order than normal chemical treatment[2]. It is important to decrease surface impurities for making diamond tools.

Table 3
Impurity content of diamond micron powder

Symbol	ANSI B74.20-1981	ГОСТ 9026-80	GB6966.1-6966.5-86	SDZ Method
M0/0.75	≤2%	≤2%+1%	≤2%	0.6%
M3.5/7	≤2%	≤2%+1%	≤2%	<0.1%
M53/75	≤2%	≤2%+1%	≤2%	<0.1%

Table 4
Size distribution of diamond micron powder

	ANSI B74.20-1981			GB6966.-6966.5-86			SDZ Method		
Symbol	large	basic	fine	large	basic	fine	large	basic	fine
M0/0.7	≤5%	≥95%	0	≤3%	≥97%	0	0.4%	99.6%	0
M3.5/7	≤5%	≥65%	≤30%	≤3%	≥79%	≤18%	0.2%	97.4%	2.4%
M53/75	≤2%	≥68%	≤30%	exceeding size range			0	98.0%	2.0%

6.Using separation ligquid No.14

In order to improve the effect of separation size of diamond grain, the separation ligquid No.14 has been designed and manufactured. Not only the precision of grain size distribution has been raised, but also the range of the products has been enlarged from M0/0.2 to M75/106.

The priciple of the sedimentation separation is Stokes' law [3]

$$V=\frac{2}{9}\frac{(\rho-\rho_1)gr^2}{\eta} \tag{3}$$

where V-grain fall velocity, r-grain half diameter of sphere shape, ρ-grain density, ρ_1-medium density, g-acceleration of gravity, η-homogeneous medium viscosity .

When the subside fall distance and the other condition has been given, we can think the fall time

$$t=k\eta/r^2 \tag{4}$$

We should indicate that η is sensitive to the temperature. It is estimated that η decreases by 2% while the room temperature increases by per 1 ℃. The operator must observe separation size condition at all time so that the fall time is regulated rationally.

7. Improving the skill for the separating operation

The siphonage devices are shown in Figure 2. General "theory" think that the liquid and the micron grain contained by it only between the up line and the down line is siphoned while the siphon is operated. In fact, that is no. To simplify, it is supposed that the siphon in the point A is very small. According to the law of momentum in the fluid mechanics, the force in the point A is as a result of the liquid pressure (Figure 3)

$$\vec{F}=-\iint_{\varepsilon}\vec{n}P\,d\sigma-\iint_{\varepsilon}\rho\vec{V}(\vec{n}\cdot\vec{V})\,d\sigma \tag{5}$$

where n-the normal line of the control surface ε, v-the fluid velocity, p-the fluid pressure in the point A, ρ-the fluid density.

Because the point A is a ditch, the relationship at the point A could be expressed as follows

$$\nabla\cdot\vec{V}<0 \tag{6}$$

where ∇-Nabla (hamiltoniam). The fluid lines are sphere symmetry and it is shown in figure 3.

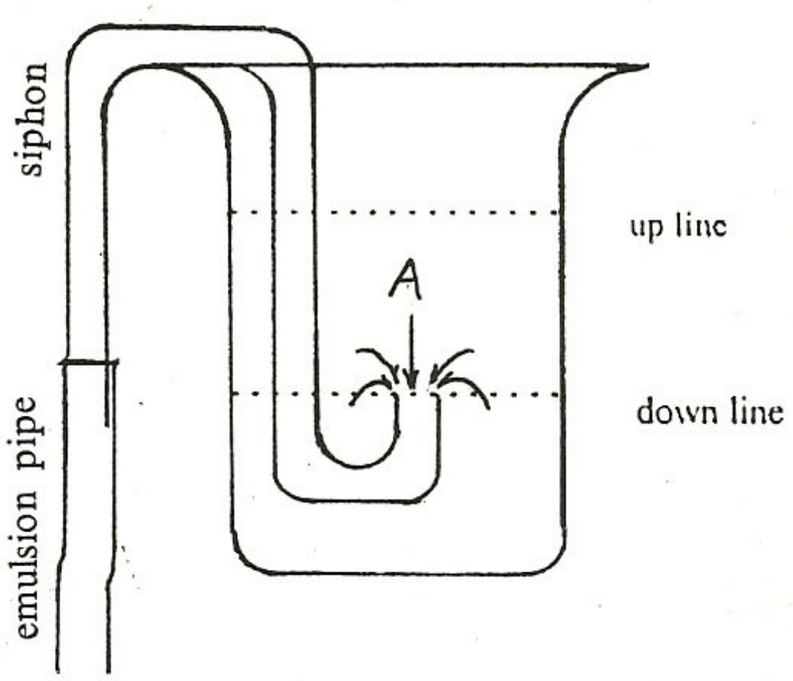


Fig . 2 The siphonage devices

It is a hinting figure of two dimension and this plane is arbitrary direction. It will be seen from this that the larger grain under the point A of the siphon entrance could been drawn out. This is one of the important reasons to disperse the product size.

In order to overcome the problem the operation skill is improved. The distance between liquid surface and the siphon end A is about 2 cm at all time while the siphoning.

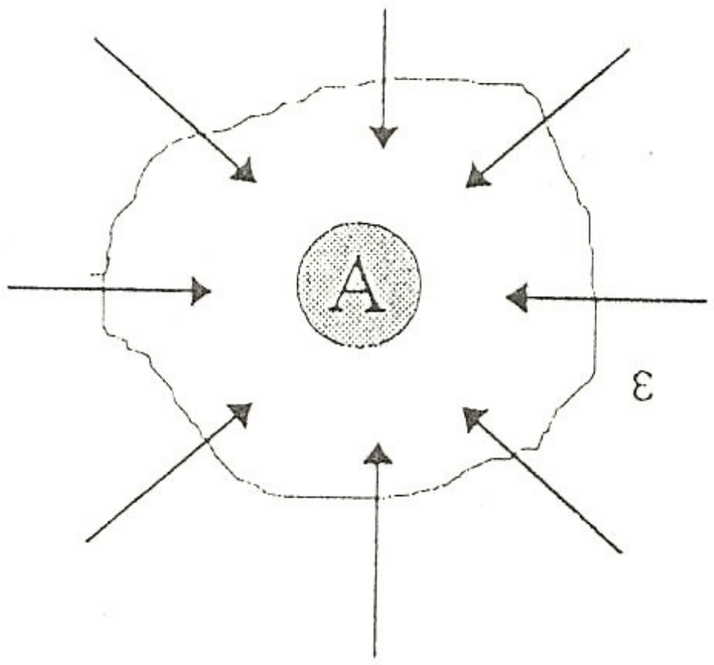


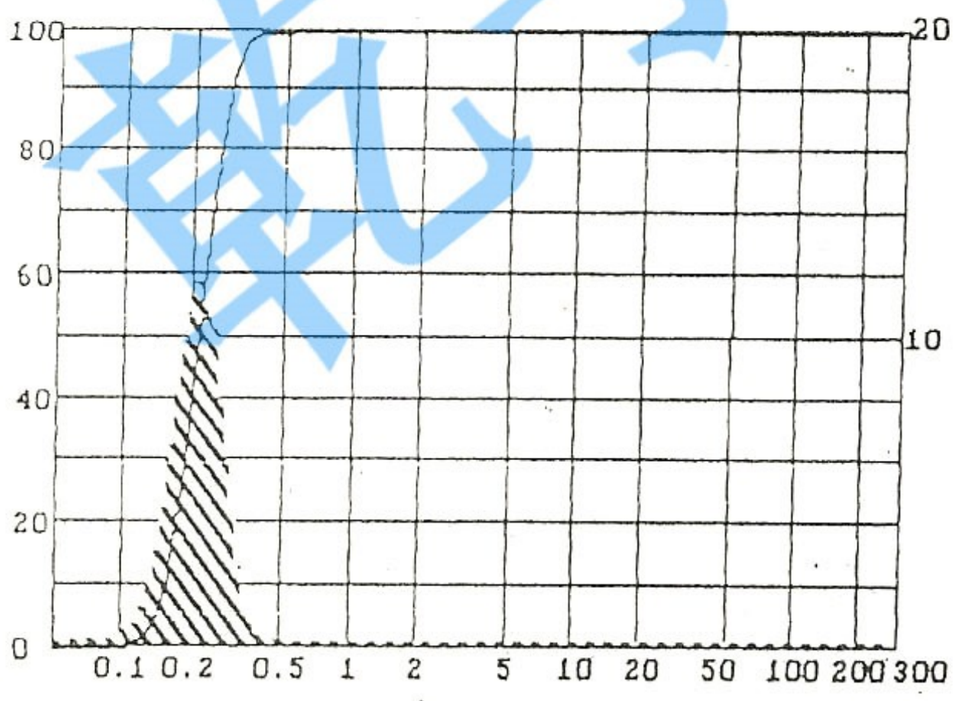
Fig . 3 The fluid lines around the end A

The value P in formula (6) depends on the high difference h between the liquid surface and the emulsion pipe end of the liquid out. According to the Torricellian law the fluid velocity in the end A

$$v = \sqrt{2gh} \qquad (7)$$

It is clear that the more h, the more V. Therefore it is not suitable for the emulsion pipe too long .and its end of the liquid out is set about 10 cm long under the end A .

After the above operation has been adopted, the value h and v has been stabilized so that the size distribution has been concentrated. It follows that the siphon times has been decreased by 1/3 and the product quality has been outstandingly increased that it could be met various need from customer .



D_{10}0.16, D_{50}0.21, D_{90}0.29, Dv0.23

Fig. 4. The size distribution of micron powder M0/0.35 made by SDZ method

It is shown in Figure 4 that particle size distribution of diamond micron powder M0/0.35 is measured by laser size analyze. The powder microscope photograph is shown in Figure 5. In both shape and size distribution, the quality of the diamond micron powder made by SDZ method has come up to advanced world standards.

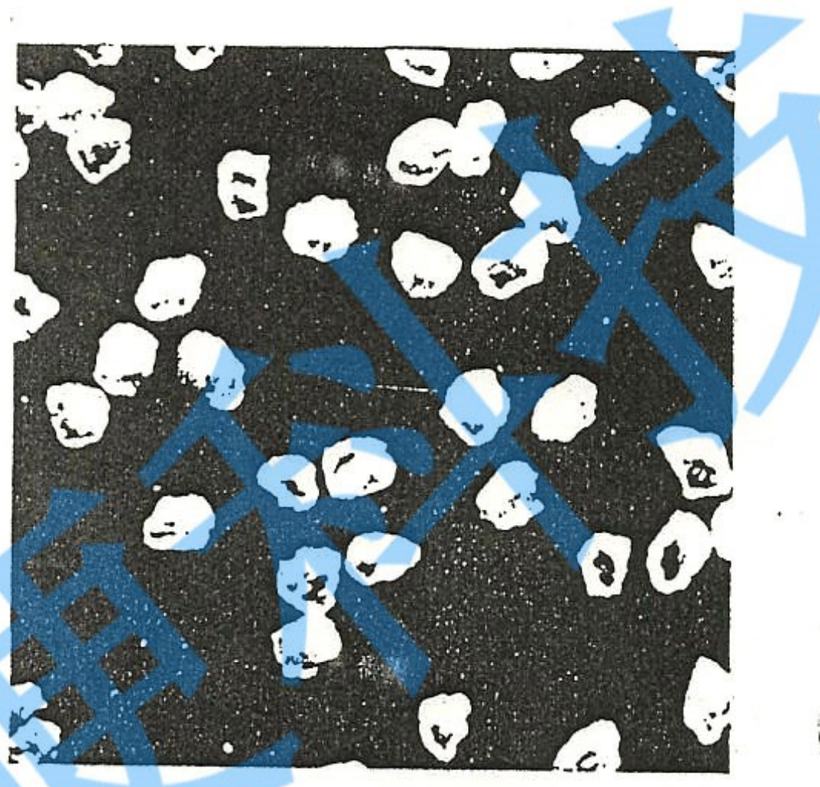


Fig.5. The photograph of micron powder made by SDZ method

8. Summary and conclusion

The main characteristics of the Pike brand diamond micron powder made by SDZ method is as follows.

1. Single grain appears regular block as well as needle and platelike have not display. Being used it, effectiveness of lapping and polishing has been raised and processed workpiece quality has been improved.

2. The powder iron-magnetism is very weak and it is beneficial to making diamond tools.

3. Total impurity content is extremely rare. Especially, impurity amount contained on grains surface is lower by one order than normal products. It is beneficial to making diamond sintered and grinding tools.

4. Many symbols of diamond micron powder more than 40μm have been manufactured by SDZ method. Its wearproof and resist impact capacity is far higher than original material.When the sintered and grinding tools are made, handle force of grains has obviously strengthened because of its rough surface. The life of these tools is lengthened and to make use of property is reformed.

References

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