Development of a Device called NSM enabling Spheroidization, Agglomeration and Surface Coating for Powder Particles

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Abstract

In this paper, we introduce a new type of powder particle shape control equipment "NSM", developed by our company, that enables spheroidization, granulation and surface coating for powder particles. NSM. Natural Graphite Spheroidization Machine, its original goal was to spheroidize the irregular shape of graphite powder particles. Graphite powder is manufactured by grinding, but its particle shape is irregular in general. If the particle shape becomes spherical by some way, the powder filling structure will be dense, leading to well electric conductivity when it is used as electrode, for example. This was the motivation of our development group, and they have challenged to develop a device, which is composed of a vertical powder processing container in which a high-speed rotor with blades is installed at the center of its bottom. As a result of the equipment operation by filling graphite powder into the container, it was confirmed that the particle shape of graphite can be spheroidized. Further, when the NSM was operated by adding a binder to the graphite powder, it was clarified that the graphite powder is granulated into the moderate particle size. The tap density of the granules is found to be improved. Furthermore, as an example, when polyethylene resin (PE) powder (particle size:100 to 200μm) and graphite powder (average particle size: about 8μm) are treated with the NSM, it is confirmed that the surface of the polyethylene (PE) particles is coated with graphite fine particles.

As described above, the NSM is an equipment for spheroidization of graphite, but from a number of subsequent experiments, it is confirmed that its capability can extend to other powders besides graphite. Thus, we appreciate very much if you could utilize the NSM for your samples' spheroidization, granulation and surface coating.

Keywords: powder processing *equipment, powder particle shape control, spheroidization of particles, granulation, compounding, particle filling structure*

1. Introduction

As for a particle shape control device, we have already developed a machine (New Gra Machine "SEG") with stirring and rolling for coating, mixing, granulation of fine particles. This machine can achieve powder coating, mixing, dispersion, granulation, etc. in one step while being simple structure and simple operability. Since the capacity of SEG is available from 1 L which is lab size to several thousand L in large one, it has been used in many powder processing fields. However, in recent years, due to the changes of materials in a global scale such as global environmental conservation, effective use of resources, and decarbonization, the miniaturization and high performance of parts have been required, and these parts are composed of powder to say the least. Accordingly, high demand has come to the powder used in such miniaturization and parts. One of the demands is particle shape control so that processing technology enabling us to do. Of course, the SEG can apply to this demand, however, it is somewhat hard to respond for the fine particle size range. The lower limit of the aiming powder particle size is several nano-meters to several micron meters, and this is the reason why our development has started. Of course, it goes without saying that conventional processing techniques for ground particle sizes remain essential, but it is necessary to develop a strategy that fully recognizes the demand for fine particle processing beyond the SEG's capabilities. Therefore, we have started the design of the fine particle shape control equipment "NSM", that can perform higher power and more effective in particle shape control processing while taking advantage of the characteristics of SEG. We have finally developed a standard equipment and its scale up version.

NSM, short for Natural Graphite Spheroidization Machine, was originally intended to spheroidization the irregular plate-like shape of graphite powder particles, as the name "graphite" suggests. Because the particle shape of the graphite powder produced by grinding is irregular so that it is not used for electrode material as it is. If it can be spheroidized in some way, the particle filling structure becomes dense, and its electric conductivity would be expected to improve. The NSM has a key device for spheroidization of fine graphite powder particles, and tested many times through in-house testing and joint research with customers. At the end, the NSM can be achieved our purpose. Not only that, it is found that this equipment has a characteristics of granulation, compounding different powders and surface coating a powder with others. In addition, it is found that this equipment enables us to cause various mechanochemical effects on the powder materials. Anyhow, the shape control of powder particles are closely related to their fluidity, granulation behavior, and surface control of particles is also influenced in physical and chemical properties of powder in both dry and wet systems.

A brief introduction on the NSM is seen from our homepage, but it is limited version. So, we have decided to have the technical report on the NSM introducing to our powder society more. We do hope that this report will inspire all of customers who are very much interested in this equipment, NSM.

2. NSM（Particle shape control device）

2.1 Basic structure of the device

Fig. 1 provides an overview of the structure of the NSM and the movement of the powder in the container due to the rotation of the agitator (standard type). That is, the appearance is the same as a general rotation processing device such as a mixer, and a motor and a cylindrical container are placed on the stage, and the rotation of the motor is transmitted to its rotation axis of the main body via a belt and pulley, and the rotor fixed to the shaft rotates to stir the powder inside the tank and performs the particle spheroidization. The biggest feature of NSM is that it has a simple structure in which the powder treatment tank has only a rotor attached to the bottom in the cylindrical container, and product recovery, subsequent cleaning in the

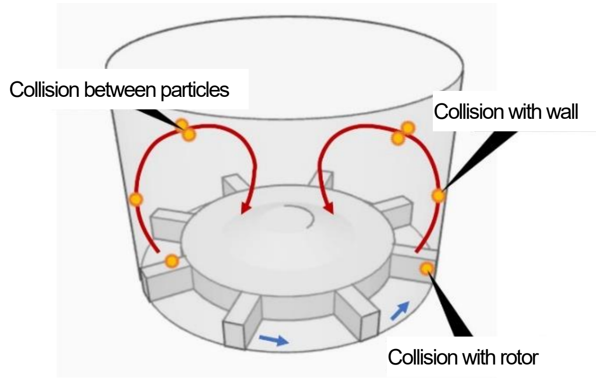


Fig. 1 The outline structure of standard NSM (bird-view) and agitator with a sketch of movement of powder in the container by the rotator in the rotator in the NSM

container, etc. are easy. In this respect, it is in contrast to the case where, for example, a chopper, a disturbing plate, a fixed blade, a circulation mechanism, a classifying rotor, or the like are installed. The features of the NSM are the rotational speed and shape of the rotor. That is, the rotor tip circumference of the NSM is designed to be three to four times larger than the SEG, up to 80 m/s. Of course, the NSM has the ability to support shaft structures and motors with high-speed rotation of agitators (rotors). In addition, the shape of the standard rotor of the NSM is designed so that the powder soaring during the operation is suitable for pushing it down and outward. By this ingenuity, strong shear and compression forces act on the powder particles in the container, so that the powder particle group is wound on the upper and outer space inside of the container, but the particles are effectively repeated collide action each other, and the movement of falling toward the rotor center direction is repeated while exercising to be rubbed against each other. The motion of this powder particles depends on the shape of the rotor and the rotational speed, and first (1), the rotation of the rotor pushes the powder to the gap between the inner wall of the tank and the tip of the rotor→ (2) the gap is affected by the swirling shearing action→ (3) Rise while turning the outer space in the container by the action of centrifugal force, face to the center of the upper lid, and → (4) fall toward the rotation center of the rotor → (5) Repeat the circulating motion to the outer space of the bottom again. During the circulation and collision motion process, the particles are spheroidized by the forces of shear and compression.

Fig. 2 shows a schematic structural of an NSM with L-shaped rotor that shows more effective spheroidization performance than the standard rotor shown in Fig.1. It is found that this L-shaped rotor makes it possible to cause the acting force of the powder particles moving from (1) to (5) described above more effective from the viewpoints of the experimental data and the DEM simulation results. That is, the residence time between the scattering of the powder wound up by the rotor rotation and its falling and scattering motion in the container is reduced, and the shear and compression forces are efficiently acted on the powder. Therefore, the L-shape rotor makes us to spheroidize the particles in a shorter time than the standard type.

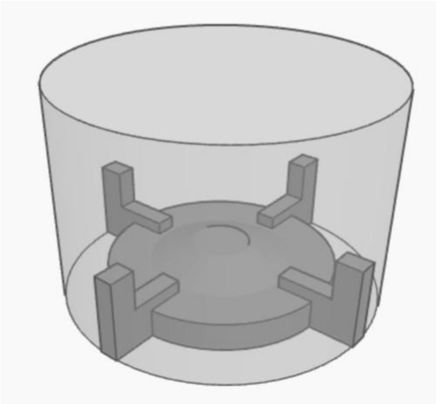


Fig. 2 Schematic structure of NSM with L-shaped rotor shape

2.2 Current NSM Line-up

Fig. 3 shows the appearance of three different NSMs that we can currently provide. Table 1 also shows the outline dimensions, capacitance, and maximum power and rotor rotation speed of these three types (200, 350 and 1000).

Fig. 3 Three types of NSM machine

Table1 NSM types and specification

Version	NSM-200	NSM-350	NSM-1000
Tank inner diameter (mm)	ϕ200	ϕ350	ϕ1000
Full capacity(L)	4	22.5	550
weight(kg)	466	600	2800
Power(kW)	11	22	90
Amount of capacity(kg)*	0.5	2.5	20
Rotational speed(rpm)	~8000	~4500	~1700

* It depends on the bulk density of the raw material and the processing content.

3. Examples by NSM

Followings are some examples introduced here, but all the shape control devices used are taken place by the NSM 350 type. The powder sample will be described in the each section.

3.1 Spheroidization of graphite powder particles by NSM (without binder)

Fig. 4 shows SEM photographs of the particles before and after treatment when the natural graphite powder is treated with the NSM-350 type. From this, the shape of the graphite particles before treatment is flaky, but after processing, it can be clearly seen that the shape looks like spherical. Note that x300 and x500 in the image of the photographs means magnifications, and these values after processing is higher than that before processing. This is to ensure that the NSM makes it possible to spheroidize the particles.

Considering the mechanism for this spheroidization of the particles with NSM, there are still many unknown points. We have done many investigations from the experimental results on particle spheroidization process causing by the NSM processing tests, visualization of the inside of the NSM container, simulation results on particle trajectory by DEM. From these investigations, it is found firstly that the particles in the container are forced by the mechanical action of shear and compression. These forces are considered to be the overall result of the direct effects of the rotors, the colliding contact of particles against other ones and with the inner walls of the container. However, it is needless to say that which force acts preferentially depends on the spheroidization of particles, but they influence surely powder particles themselves in the container. In other case, if the powder material is soft, the forces will act on it, then the powder material spheroidization gradually proceeds with plastic deformation. On the contrary, if the

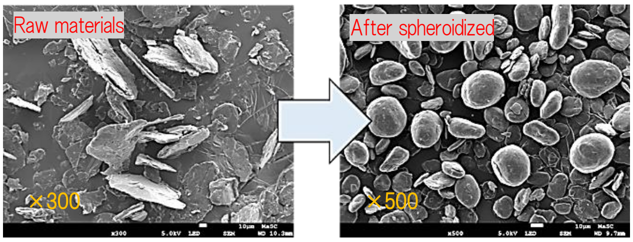


Fig. 4 SEM photos of the graphite particles before and after treatment by the NSM-350 (left: before, right: after treatment)

particles are hard and brittle, there are few deformations during the operation, but microcracks create inside and surface of the particles due to the action of surface fracture and repeated stress during the operation. This microcracks make it possible to cause plastic deformation of the particles, at which plastic deformation starts with increasing its plastic strain energy stored in the particles. This energy is finally released and transferred into mechanochemical effects. Thus even though a hard and ductile material receives such repeated mechanical action, then its fracture mode is changed into ductile one, and this is the reason why surface coating and surface tipping of particles is accelerated in the presence of other fine powder in the milling operation. This phenomenon is similarly happened by the treatment of NSM. Fig.5 is a verification example of these considerations. In this figure, SEM photographs of PTFE particles treated by the NSM are shown. PTFE means poly-tetrafluoro ethylene (PTFE) as a representative of soft materials. Since the result when the graphite, a brittle material, is treated with NSM has already been shown in Fig. 4. Referring to it, as a result under another condition, Fig. 6 shows the situation of spheroidization that seems to be the result of pulverization by shear force on the particle surface in the treatment process. From these photographs, it can be confirmed that the particle shape is spheroidized by the NSM treatment of the mixture of PTFE and graphite powders.

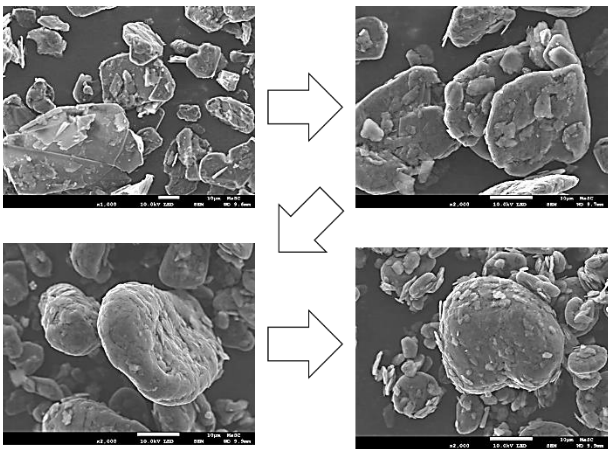


Fig. 5 SEM photos of the particle shape before and after treatment of PTFE powder by the NSM-350

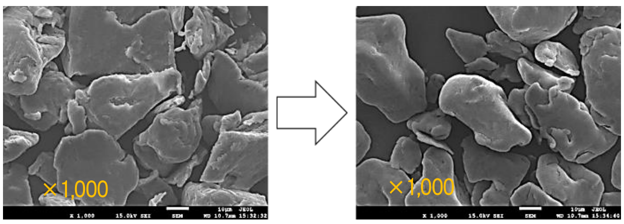


Fig. 6 SEM photos of the graphite particles before and after treatment by the NSM-350

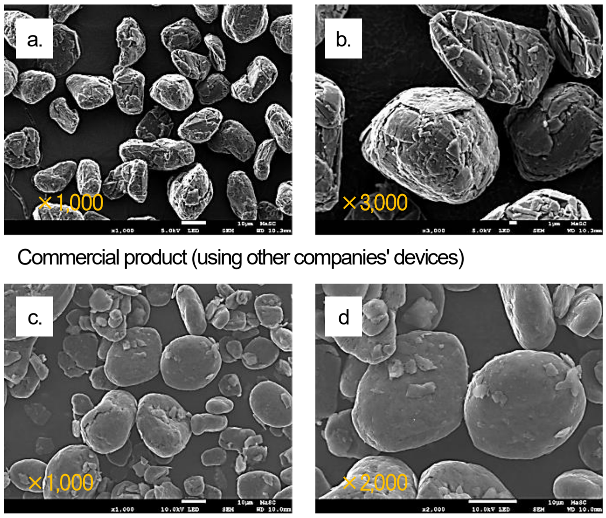


Fig.7 SEM photos of the sample (D50=17-18μm) a, b, provided by a third part's spheroidization device with those (D50=20μm) c, d prepared by the NSM-350 type

Fig. 7 intends to compare SEM photographs, a, b (about D50: 20 μm) of the graphite particles commercially available prepared by the third part device with those of the particles, c, d treated by the NSM-350 type. From the viewpoint of spheroidization of these particles, the particle surface treated by the NSM is relatively smooth and its spheroidization looks to be effective. The measurement results on the tap density of both samples are as follows.

Tap density of spherical graphite particles by third-party equipment＝0.97g/cc

Tap density of spherical graphite particles by NSM＝1.18g/cc

Here, the tap density of the graphite powder was measured as its spheroidization by the NSM. In general, tap density of a powder is important characteristic, and especially for the graphite powder, it is often used as a negative pole material of secondary batteries. That is, the material is ground by a mill, then the shape of the particles is flaky and irregular. Such ground graphite powder is not used as it is because its tap density is not good, so that it is desirable to change the shape of particles into round and spheroidized one in order to improve tap density and suppress anisotropy to the electrical conductivity of graphite particles (bringing them closer to the isotropic powder characteristics). This treatment can improve the energy density of secondary batteries and reducing expansion and shrinkage of the electrode during its charging and discharging cycles. For example, grinding graphite is commonly taken place until about 10 μm in its average particle size, then it is normally to spheroidizing treatment and classification operation by some way. Apart from the details here, the results of once the graphite raw material powder is ground, then spheroidized after classification to cut off certain particle size range are introduced as follows. At first, the graphite raw materials are ground with our mechanical grinder, and the average particle size is about 12 μm. This powder was subjected to spheroidization by the NSM, followed by classification. Fig. 8 shows SEM photographs of the shape of each powder particle when it is spheroidized with NSM and then classified, and the particle size distribution of these powders is shown in Fig.9. Furthermore, Table 2 shows the experimental results on the average particle size (D50 [μm]) and the tap density [g/cc] of the each powders.

From these results, it can be seen that the tap density (0.88g/cc) of the particles by the spheroidization is greater than that (0.52g/ cc) of the ground product, and the value of the tap density changes at classification size, but the value is further improved by cutting (removing) the fine powder.

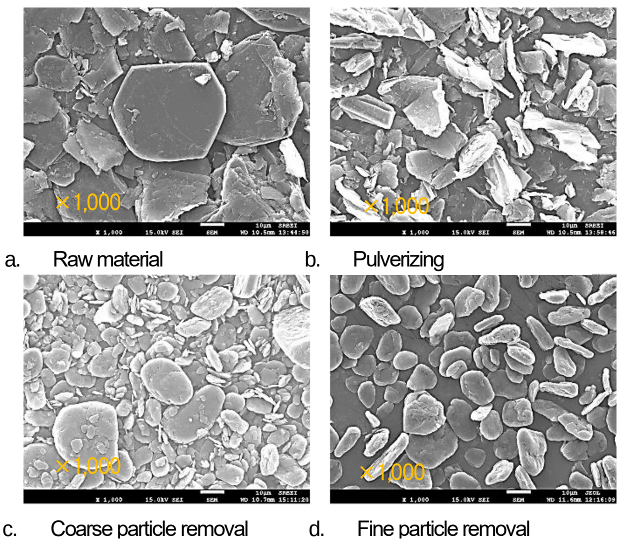


Fig. 8 SEM photos of the raw graphite particles a and the ground one, b, the ones after treatment by the NSM c, and the particles classified into certain size range, d

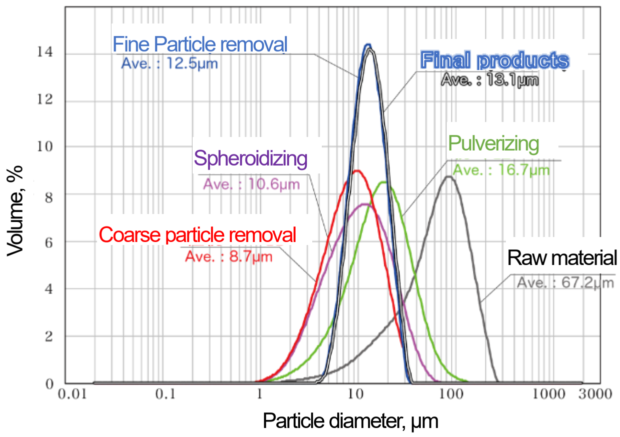


Fig. 9 Particle size distribution changes of the graphite particles after grinding, spheroidization, and classification

Table 2 D50 and tap density of graphite particles treated under different operations

Operation	D50 [μm]	Tap density [g/cc]
Raw material	67.2	0.90
Grinding	16.7	0.52
Spheroidizing	10.6	0.88
Coarse particle removal	8.7	0.78
Fine particle removal	12.5	1.06

3.2 Spheroidization of graphite particles when binders are added to them in the operation by NSM

Of course, grinding process of the graphite raw materials is necessary, but this operation needs a lot of energy besides the fact that the product yield is low due to the generation of fine powder. Therefore, from the viewpoint of reducing the overall cost, the raw material is pulverized by the NSM performing the spheroidization and pulverization. Now we have started this operation from the raw graphite powder (average diameter = about 150 μm) with the NSM, and the binder is mixed in the additional NSM treatment, as follows.

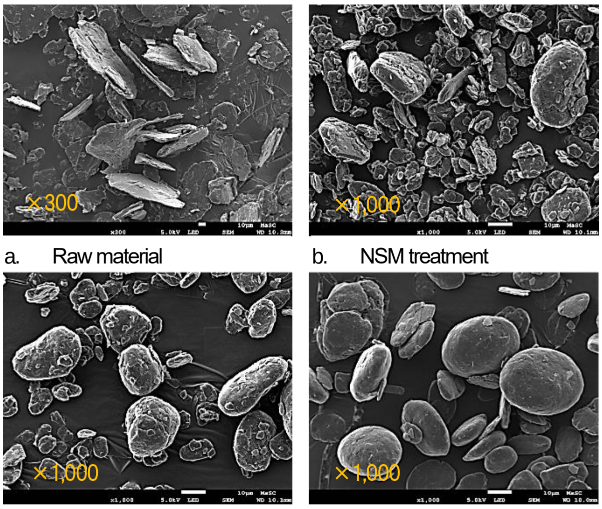
Table 3 shows the average diameter (D50) and the tap density of the graphite powders at different stages. That is, these stages are four, i.e., the raw material at that time, after the spheroidization by the NSM and after the additional treatments by the NSM by adding a water and a polymer binder. It is noteworthy that the tap density of the particles after the spheroidization without binder becomes larger than that of the starting sample (ground one) even though the particle size has been reduced due to the grinding and the particles spheroidizing phenomena at the same time. As can be seen from the results of Table 2, shown earlier, the tap density of the graphite ground decreases as the particle size decreases. Therefore, the NSM has the potential of two things proceed simultaneously; one is surface grinding of particles and the other is spheroidization of the particles. In these phenomena, the fine powder are produced, and they are agglomerated with increasing the amount of the particles and sizes. Fig.10 shows SEM photos of the raw material and the processed products with binder. From the figure b, it is clearly found that the particles look like round shape and become thick by the treatment, and the granulation effect of fine particles is demonstrated in this process. Furthermore, from the SEM image shown in Fig. 10 c, the particles look like spherical shape, this may be due to the spheroidization by using a water-based binder. Similarly, Fig.10 d looks like also round shape ones, and this is also suggested for the particles to be spheroidized by using a polymeric binder. Of course, it is noted that the appearance and properties of the spheroidized particles differ depending on the type of binder and the operational condition of the NSM.

Table 3 D50 and tap density of the powder particles for the raw graphite particles, the NSM treatment, additional spheroidization by the NSM with water and polymer base binders respectively

Operation	D50 [μm]	Tap density [g/cc]
Raw graphite (-100 mesh)	59.5	0.80
NSM treatment	9.7	1.05
Additional operation with water base binder	12.4	1.12
Additional operation with polymer base binder	23.2	1.21

3.3 Granulation of graphite powder by NSM

The purpose of agglomeration/granulation of natural graphite is to improve fluidity and handling and add isotropic to the graphite powder. Here, an example of the granulation by the NSM is shown below: The average particle size of graphite raw material powder is 17μm, but the tap density is as low as 0.14g/cc. Table 4 shows the results of granulation of this raw material powder treated under various conditions using the NSM-350 type. By changing the operational conditions, the particle size of the granulation can be adjusted at about 50 to 200 μm, and its tap density is also changed from 0.58 to 0.78.



a. Raw material
b. NSM treatment
c. Additional operation with water base binder
d. Additional operation with polymer base binder

Fig. 10 SEM photos of the natural graphite raw material powder a, the sample after spheroidization with NSM, b and the sample for the additional operation by the NSM with water binder, c, and that with polymeric, d

Table 4 D50 and tap density of the graphite powders after granulation with different conditions

Specification	D50 [μm]	Tap density [g/cc]
Raw graphite	17	0.14
After granulation Condition-1	46	0.58
After granulation Condition 2	157	0.63
After granulation Condition 3	201	0.78
After granulation Condition 4	133	0.62

Fig. 11 shows SEM photos of graphite granules, but this granules shape is different from the graphite product shown in the paragraphs 3.1 to 3.2, and in particular, the granule sizes seem to increase in the direction of depth in comparison with those of the spherical products, and appear to be closer to the sphere. On the other hand, there is no smoothness of the surface in comparison to the spherical products. In addition, it is necessary to measure particle density, but it also appears that the density of particles is low from SEM images. It should be noted that the results shown here are not data under optimal conditions. In any case, the NSM has ability of spheroidization and granulation/agglomeration by controlling the rotor speed and the operational condition.

All in all, various particle shape products can be produced by controlling the operational condition of the NSM.

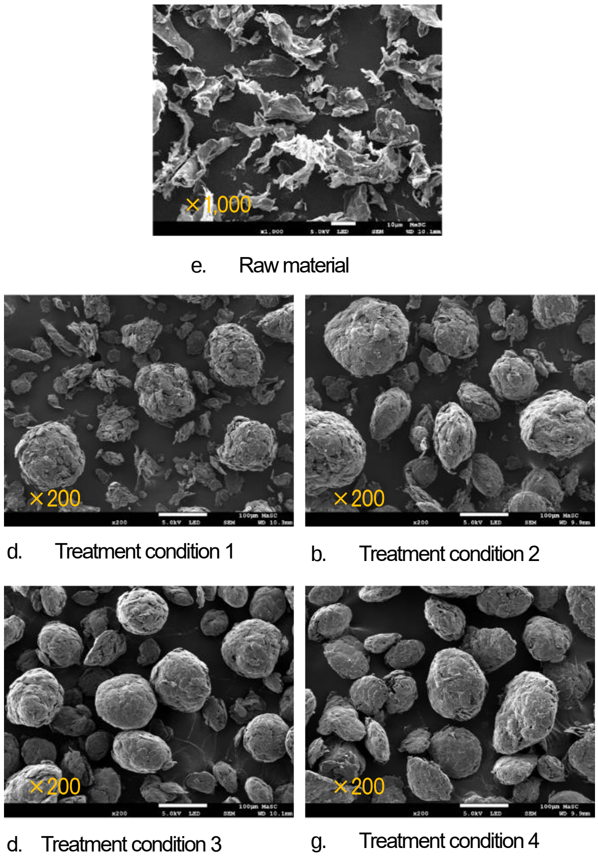


Fig.11 SEM photos of the graphite powder (flake particles) raw material a, and granulation processed products by the NSM-350 type (treatment conditions 1 to 4) b to e

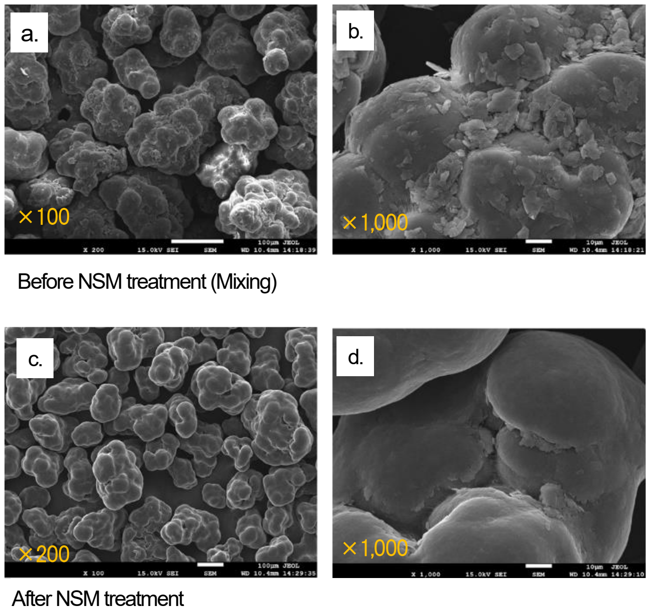


Fig.12 SEM image of the graphite and PE powders mixed, a and b, the mixtures processed by the NSM, c, d

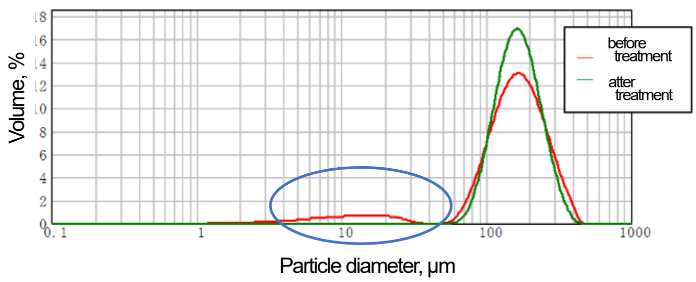


Fig.13 Size distribution curves of the PE and graphite mixtures before and after treatment by the NSM

3.4 Coating resin powder particles with graphite fine particles (particle design)

"Graphite coating" means particles of a material coated with graphite powder. As a mother particles surface is coated with fine guest particles, the surface property of the coated particles is modified by the guest ones. In general, the purpose of coating particles surface with guest particles is to modify and change with the guest particles, for example, such coating makes it possible to improve electric conductivity, thermal conductivity, physical and mechanical surface properties such as tap density and rust protection. Hereinafter, an example of coating polyethylene (PE) resin powder with natural graphite fine particles is introduced.

Fig. 12 shows SEM photos of the PE powder particles with 100 to 200μm coated with graphite fine particles (D50= about 8μm). That is, a in the figure is the mixture simply prepared by mixing the two kinds of powder, b shows the same mixture, but different enlarged screen, c and d are the mixture treated by the NSM-350 at different enlarged screens. From this figure, it is seen that many fine particles of graphite adhere on the surface of the PE particles by the simple mixing operation, but the surface of the particles treated by the NSM looks like smooth and it is hard to distinguish the difference in particle size and shape of the graphite particles there. This implies that no more fine particles are left in the treatment by the NSM, and they may be embedded in the surface of the PE particles.

Fig.13 shows the particle size distributions of the mixture of the graphite fine powder and the PE powder. From this figure, the peak derived from the graphite fine powder (below 30μm) after the NSM treatment is found to disappear, while these fine particles are remained for the untreated sample. Reconsidering the fact of Fig.12, it can be concluded that the graphite fine particles were composited to the PE particle surfaces. This is just an example of compounding the PE particles of 100-200μm with the graphite ones of 8μm by the NSM, but this machine is applicable for further fine particle mixture composing of mother particles and guest ones, each size of submicron to nano-size order. As a proof, Fig.14 shows SEM photos of starting and end products treated by the NSM. The mixture is a composite of polypropylene (PP) particles with 3μm in average particle size and carbon black with 2 nm in primary particle size. From this figure, it can be seen that the PP particle surface after treatment by the NSM is surely coated with the carbon black, and it looks like uniform.

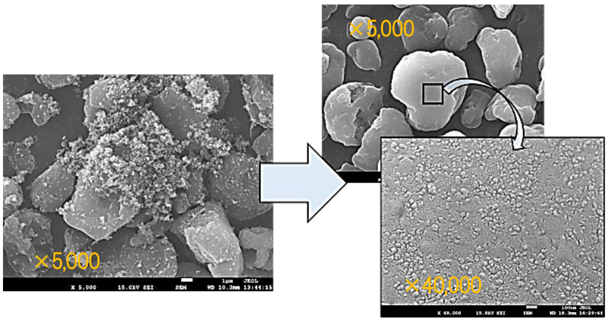


Fig.14 SEM photos of the mixtures of PP particles (D50=3μm) and carbon black (D50=20nm) before and after treatment by the NSM

4. Concluding remarks

The NSM is the device enabling us to process various particle designs such as spheroidization, agglomeration/granulation and surface coating. This paper introduces some of the ability of the NSM together with the facts on mainly graphite powder that this powder particles become spherical or cause agglomeration/granulation phenomena when the material itself or mixed with others are treated by the NSM. It is well known that natural graphite is used as a pencil core, however not only this, but also in recent years, it is very useful for heat dissipation materials in secondary batteries, conductive additives, electronic devices and other properties. Accordingly, there will be increase in the chance of use for graphene, and its use form will be definitely compounding such as spheroidization, agglomeration/granulation and surface coating.

Due to mainly confidentiality with our clients, further details such as operational condition and materials in the NSM processing are unable to fully introduce here, but according to our list, there are more than 50 companies tested using the NSM series, more than 60 materials, and more than 1000 test scores. We have decided to introduce our unique machine of NSM to powder technology and processing fields who are very much interested in. In addition, we are always making an effort to improve this machine's ability and performance more so as to meet the requests as much as possible. No matter how the industry changes in the future, we will be able to respond immediately to all customers/clients and to continue the NSM be loved for many years.

URL : https://www.betterseishin.co.jp/english/product/plant/nsm.html

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