

COMPOSITE POWDER CONTAINING YTTRIA AND METHOD FOR
MANUFACTURING SAME

CROSS-REFERENCE TO RELATED APPLICATION(S)

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This application is based on and claims priority to Korean Patent Application No. 10-2024-0181511, filed on December 9, 2024, in the Korean Intellectual Property Office, the disclosure of which is herein incorporated
10 by reference in its entirety.

BACKGROUND OF THE INVENTION

1. Field of the invention

15 The present disclosure relates to a composite powder containing yttria and a method for manufacturing the same and, more specifically, to a composite powder having plasma etching resistance, which can be used in coating processes, such as thermal spray coating and aerosol
20 deposition, and a method for manufacturing the same.

2. Description of the Prior Art

High-integration and ultra-fine line width techniques in semiconductor processes require plasma
25 etching under extreme environment conditions, such as

high-density plasma, high cleanliness, and excessive electric shocks. In particular, plasma etching using reactant gases containing a halogen element with strong chemical reactivity, such as F, Cl, or Br, involve
5 etching of various deposition materials on the wafer surface, but provoke chemical and physical reactions with metal or ceramic components inside a chamber, thereby causing damage to the surface of the components and generation of non-volatile contaminant particles.

10 These contaminant particles critically affect the yield of final semiconductor devices, and therefore, components of plasma-resistant materials for minimizing the generation of contaminant particles are required. In recent years, interest has been greatly increasing in
15 techniques for coating the surfaces of metal or ceramic components with ceramic materials exhibiting excellent plasma etching resistance. Typically, yttria or yttrium oxide (Y_2O_3) coating is widely applied.

Yttrium oxide exhibits a high melting point
20 ($2,450^{\circ}C$), chemical stability, and crystallographic stability up to $2,300^{\circ}C$. Particularly, yttrium oxide exhibits excellent plasma resistance due to excellent chemical stability against F radicals, high ion-impact resistance resulting from the high atomic mass of yttrium,
25 and excellent mechanical properties of the reaction

product YF_3 .

However, the reaction of the upper surface of a yttrium oxide coating layer with plasma gas, such as SF_6 , CF_4 , CHF_3 , or HF at the initial stage of the etching
5 process causes a change in the concentration of fluorine-based gas within the chamber. Consequently, the seasoning time in the etching process increases, and the yttrium oxide surface and the plasma gas react to form contaminant particles containing fluorine. Moreover,
10 when yttrium oxide undergoes thermal cycling, stress arises due to a difference in the coefficient of thermal expansion between the contaminant particles and yttrium oxide, causing the detachment of the contaminant particles.

15 To address these problems, YF_3 with excellent corrosion resistance has been introduced, but YF_3 melts in the ultra-high-temperature plasma during atmospheric plasma spraying (APS), and a part of a fluoride is oxidized, resulting in a coating layer in which the
20 fluoride and the oxide are partially mixed. Moreover, such a coating layer, compared with yttrium oxide spray-coated layers, may cause problems, such as cracking within the coating layer and frequent generation of contaminant particles in the etching chamber.

25 Meanwhile, the plasma resistance of plasma-

resistant members depends on the grain size, and thus, as the grain size is larger, the roughness change after plasma etching is greater, causing reduced plasma resistance. Moreover, the larger the grain size, the
5 larger the contaminant particles generated by plasma etching. Large contaminant particles cannot exit the chamber by fluid flow and are thus adsorbed onto the wafer, thereby hindering the operation of semiconductor devices and reducing yield. Therefore, a need exists for
10 thermal spray coating of yttrium-oxide-based compounds having small particle sizes.

To prevent such contaminant particles, an attempt has been made to control the microstructure of a coating material. In sintered bodies produced by mixing yttrium
15 oxide and magnesium oxide (MgO), two types of insoluble composites are formed and thus suppress each other's grain growth due to a mutual pinning effect. The sintered bodies with suppressed grain growth has smaller grains than conventional yttrium oxide sintered bodies,
20 resulting in reduced surface roughness. Therefore, the generation of contaminant particles is reduced during plasma etching. However, when components in the chamber are manufactured from yttrium oxide or yttrium-oxide composite sintered bodies, process stability is degraded
25 due to the brittleness of ceramic materials.

On the other hand, the hardness of plasma-sprayed coatings affects semiconductor process yield. A low coating hardness may result in the generation of contaminant particles due to plasma impact or high
5 temperatures. Therefore, there is a need for thermal spray coating of high-hardness yttrium-oxide-based compounds.

SUMMARY OF THE INVENTION

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An aspect of the present disclosure is to provide a yttria-magnesia composite powder with plasma etching resistance, which can be used in coating processes, such as thermal spray coating and aerosol deposition.

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In accordance with an aspect of the present disclosure, there is provided a method for manufacturing a composite powder, the method including: preparing a slurry in which a yttria powder, a magnesia powder, a solvent, and an organic additive are mixed; performing
20 ball milling on the slurry; and drying the slurry to produce a granular powder containing the yttria powder and the magnesia powder, wherein the granular powder contains yttria crystals and magnesia crystals.

The slurry may be spray-dried to produce the
25 granular powder.

The organic additive may be contained in an amount of 1 to 6 wt% relative to the total weight of the yttria powder and the magnesia powder.

5 The organic additive may contain a dispersant and a binder, and relative to 100 wt% of the organic additive, the dispersant may be contained in an amount of 30 to 90 wt% and the binder may be contained in an amount of 10 to 70 wt%.

10 The binder may be at least one of PVA, PVB, PVP, and PEO.

The RIR of the yttria powder to the magnesia powder contained in the granular powder may be 30:70 to 80:20.

The RIR of the yttria powder to the magnesia powder contained in the granular powder may be 35:65 to 79:21.

15 Relative to a total of 100 wt% of the yttria powder, the magnesia powder, and the solvent, the sum of the yttria powder and the magnesia powder may be 30 to 70 wt%.

20 In the granular powder, relative to a total of 100 vol% of the yttria powder and the magnesia powder, the volume ratio (vol%) of the yttria powder to the magnesia powder may be 30:70 to 70:30.

In accordance with another aspect of the present disclosure, there is provided a composite granular powder,
25 containing a yttria powder and a magnesia powder, wherein

the volume ratio (vol%) of the yttria powder to the
magnesia powder is 30:70 to 70:30.

The composite granular powder may be produced by
spray-drying a slurry in which the yttria powder, the
5 magnesia powder, a solvent, and an organic additive are
mixed.

The RIR of the yttria powder to the magnesia powder
contained in the granular powder may be 30:70 to 80:20.

The RIR of the yttria powder to the magnesia powder
10 contained in the granular powder may be 35:65 to 79:21.

According to embodiments of the present disclosure,
contaminant particles within a chamber can be reduced
by micronizing the crystalline phase.

Furthermore, according to embodiments of the present
15 disclosure, the strength of the coating layer can be
enhanced and the manufacturing cost can be reduced.

Furthermore, according to embodiments of the present
disclosure, a coating having excellent plasma etching
resistance can be formed on a component with a complex
20 shape.

BRIEF DESCRIPTION OF THE DRAWINGS

The above and other aspects, features and advantages
of the present disclosure will be more apparent from the
25 following detailed description taken in conjunction with

the accompanying drawings.

FIG. 1 shows a flow chart illustrating a method for manufacturing a composite powder according to an embodiment of the present disclosure.

5 FIGs. 2A to 2G show SEM images of the granule surfaces according to examples and comparative examples of the present disclosure.

FIG. 3 shows EDS images of a granule according to an example of the present disclosure.

10 FIGs. 4A to 4G show graphs illustrating the XRD analysis results of the granular powders according to examples and comparative examples of the present disclosure.

15 FIGs. 5A to 5G show graphs illustrating the PSA analysis results.

FIGs. 6A to 6F show SEM images of coating layer sections for examples and comparative examples of the present disclosure.

20 FIGs. 7A and 7B illustrate the grain size distributions for Comparative Example 1 and Example 2.

FIGs. 8A and 8B illustrate crystalline structures for Comparative Example 1 and Example 2.

FIGs. 9A and 9B illustrate the crystallographic orientations for Comparative Example 1 and Example 2.

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DETAILED DESCRIPTION OF THE EXEMPLARY EMBODIMENTS

Hereinafter, embodiments of the present disclosure are described in detail with reference to the accompanying drawings, and similar or identical elements are assigned the same reference numerals irrespective of figure numbers, and redundant descriptions thereof are omitted. In the following description of the embodiments, it will be understood that, when a layer (or film), a region, a pattern, or a structure is referred to as being "on" or "under" another substrate, another layer (or film), another region, another pad, or another pattern, it can be "directly" or "indirectly" on the other substrate, layer (or film), region, pad, or pattern, or one or more intervening layers may also be present. Such a position of the layer has been described with reference to the drawings.

Additionally, the top/above, bottom/below, left/left side, right/right side, vertical (up/down), and horizontal (left/right) of each floor will be described with reference to the drawings. In the drawings, the thickness or size of each layer is exaggerated, omitted, or schematically illustrated for convenience and clearness of description. In addition, the size of each element does not fully represent its actual size.

It should be understood that the terms "comprise",
"include", "composed of", and the like herein specify
certain features, numbers, steps, operations, elements,
or some or combinations thereof, but do not preclude the
5 presence or possibility of one or more other features,
numbers, steps, operations, elements, or some or
combinations thereof in addition to the description.

The terms first, second, and the like may be used
herein to describe various elements. These elements
10 should not be limited by these terms, as these terms are
only used to distinguish one element from another unless
stated otherwise or the context clearly indicates
otherwise.

The term "about" or the like may indicate the usual
15 error margin for each value, which is readily known to a
person skilled in the art, and the term "about" or the
like may indicate $\pm 0.5\%$ or up to 1% of the indicated
value. Additionally, the term "about" or the like may
indicate a measurement error due to the limitations of
20 the measuring method.

In the description of embodiments herein, a detailed
description of known techniques associated with the
present disclosure may be omitted to avoid unnecessarily
obscuring the subject matter of the present disclosure.

25 The accompanying drawings are intended to facilitate

the understanding of embodiments herein, and the technical spirit disclosed herein is not limited by the accompanying drawings, and rather should be construed as including all the modifications, equivalents and
5 substitutes within the spirit and technical scope of the present disclosure.

Hereinafter, embodiments of the present disclosure will be described with reference to the accompanying drawings.

10 FIG. 1 shows a flow chart illustrating a method for manufacturing a composite powder according to an embodiment of the present disclosure.

Referring to FIG. 1, a method for manufacturing a composite powder according to an embodiment of the
15 present disclosure includes: a step of preparing a slurry in which an yttria powder, a magnesia powder, a solvent, and an organic additive are mixed (S100); a step of performing ball milling on the slurry (S110); and a step of drying the slurry to produce a granular powder
20 containing the yttria powder and the magnesia powder (S120). Particularly, relative to a total of 100 wt% of the yttria powder, the magnesia powder, and the solvent, the sum of the yttria powder and the magnesia powder may be 30 to 70 wt%. Additionally, in the granular powder,
25 relative to a total of 100 vol% of the yttria powder and

the magnesia powder, the volume ratio (vol%) of the yttria powder to the magnesia powder may be 30:70 to 70:30.

5 The composite powder according to an embodiment of the present disclosure may be a composite powder containing yttria (or yttrium oxide, Y_2O_3) and magnesia (or magnesium oxide, MgO). The composite powder according to an embodiment of the present disclosure may be preferably a granular powder.

10 In an embodiment of the present disclosure, for example, a composite granular powder in which the yttria and magnesia powders are mixed at a volume ratio (vol%) of 30:70 to 70:30 may be manufactured using spray drying. If the volume fraction of either yttria or magnesia is
15 below 30 vol% or above 70 wt%, the insoluble composite granular powder thus manufactured may fail to exert a mutual pinning effect. The absence of the mutual pinning effect results in yttria and magnesia being unable to suppress each other's grain growth, thereby failing to
20 achieve microstructure control. Consequently, the material occupying the larger volume between yttria and magnesia undergoes grain growth, causing an increase in the grain size of the composite granular powder.

Hereinafter, the method for manufacturing a
25 composite powder according to the embodiment of the

present disclosure will be described in detail.

To manufacture a composite granular powder by spray drying, a slurry is prepared as below.

The slurry is prepared by adding a total of 30 to 5 70 wt% of yttria and magnesia powders and 30 to 70 wt% of a solvent (ethanol), relative to 100 wt% of the slurry excluding an organic additive. With respect to the ratio of the yttria powder and the magnesia powder, the yttria powder in an amount of 30 to 70 vol% and the magnesia 10 powder in an amount of 70 to 30 vol% are added relative to a total of 100 vol% of the powder volume. The organic additive is added in an amount of 1 to 6 wt% of the total weight of the two ingredients, the yttria powder and the magnesia powder. In 100 wt% of the organic additive, 30 15 to 90 wt% of a dispersant and 10 to 70 wt% of a binder are contained.

If either of the yttria or magnesia powder is below 30 vol% or above 70 wt%, the insoluble composite granular powder thus manufactured may fail to exert a mutual 20 pinning effect. The yttria powder is used in the form of cubic-shaped particles with a particle size of 3 to 5 μm , and the magnesia powder is used in the form of cubic-shaped particles with a particle size of 0.1 to 1 μm . In an embodiment of the present disclosure, the yttria 25 powder and/or the magnesia powder may be a primary powder

or primary powders.

The organic additive, when added in an amount below the value described above, may result in decreased granule strength, thereby affecting heat treatment, a subsequent process and increasing the amount of fine particles. The organic additive, when added in an amount above the value described above, may result in increased slurry viscosity, thereby posing difficulty in slurry addition and causing non-uniform granule shapes.

The dispersant is added to facilitate the dispersion of each ingredient and to provide viscosity for ensuring uniformity during the formation of a coating layer, and at least one of a polyacrylate salt, polymethacrylic acid, polycarboxylic acid, and an ether may be used as the dispersant. The dispersant, when added in an amount below 30 wt%, may exhibit an insignificant effect, whereas the dispersant, when added in an amount above 90 wt%, may reduce the packing density of granules and cause non-uniform granule shapes.

The binder is added to enhance the adhesion between granules during press-molding of the granules, thereby imparting strength to molded bodies, and at least one of PVA, PVB, PVP, and PEO may be used as the binder. The binders, such as PVA, PVB, PVP, and PEO, exhibit excellent compatibility with the solvent and the powders.

Additionally, such binders are also superior in terms of slurry stability including viscosity and solubility. The binder, when added in an amount below 10 wt%, may cause difficulty in strength enhancement, whereas the binder,
5 when added in an amount above 70 wt%, may increase the viscosity of the slurry, causing defects during spray drying.

A ball milling process is performed at 200 to 350 rpm for 40 hours or shorter.

10 The slurry thus prepared is spray-dried to manufacture granules. The granulation may be a process of forming a powder mixture into agglomerates. In an example of the present disclosure, a rotary atomizer was used as a spray-drying apparatus, but the present
15 disclosure is not limited thereto.

The spray-drying apparatus was set with an inlet temperature of 80-120°C and a rotary atomizer disk speed of 3,000-30,000 RPM.

If the inlet temperature of the spray-drying
20 apparatus is below 80°C, granules may not be properly formed, and sufficient drying may not be achieved, causing granules to stick together, forming agglomerates, or causing incompletely dried granules to adhere to the chamber walls of the atomizer. If the inlet temperature
25 of the spray-drying apparatus is above 120°C, granules

may have non-uniform shapes or non-spherical grains may be formed.

If the rotation speed of the rotary atomizer disk (atomizer speed) is below 3,000 RPM, large droplets may be generated due to a low rotation speed, and thus are not sufficiently dried in the chamber, whereas if the rotation speed of the rotary atomizer is above 30,000 RPM, fine grains may be generated, resulting in a reduction in yield.

In an embodiment of the present disclosure, the inlet temperature is preferably about 100°C, and the rotation speed of the rotary atomizer disk is preferably about 12,500 RPM. Through the spray drying according to an embodiment of the present disclosure, granules with a D50 of 30 to 40 µm were manufactured.

[Experimental Examples]

Table 1 shows mixing ratios of the primary powders.

TABLE 1

Mixed Powder	Ingredient	Mixing ratio (vol%) of primary powders
Preparation Example 1	Y ₂ O ₃	100
Preparation Example 2	Y ₂ O ₃	99
	MgO	1
Preparation Example 3	Y ₂ O ₃	90
	MgO	10
Preparation	Y ₂ O ₃	70

Example 4	MgO	30
Preparation	Y ₂ O ₃	50
Example 5	MgO	50
Preparation	Y ₂ O ₃	30
Example 6	MgO	70
Preparation	Y ₂ O ₃	10
Example 7	MgO	90

Each of the prepared mixed powders in Table 1 was subjected to spray drying. Table 2 shows spray process conditions for the examples and comparative examples of the present disclosure.

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TABLE 2

Classification	Mixed powder	Atomizer rotation speed (RPM)	Milling time	Inlet temperature (°C)
Comparative Example 1	Preparation Example 1	12,500	20	100
Comparative Example 2	Preparation Example 2	12,500	20	100
Comparative Example 3	Preparation Example 3	12,500	20	100
Example 1	Preparation Example 4	12,500	20	100
Example 2	Preparation Example 5	12,500	20	100
Example 3	Preparation Example 6	12,500	20	100
Comparative Example 4	Preparation Example 7	12,500	20	100

FIGs. 2A to 2G show SEM images of the granule surfaces according to the examples and comparative examples of the present disclosure.

10 FIG. 2A shows the granule surface image of Comparative Example 1, FIG. 2B of Comparative Example 2,

FIG. 2C of Comparative Example 3, FIG. 2D of Example 1,
FIG. 2E of Example 2, FIG. 2F of Example 3, and FIG. 2G
of Comparative Example 4. FIGs. 2A to 2G indicate field
emission scanning electron microscope (FE-SEM) images,
5 and JSM-IT700HR model (JEOL) was used. The magnification
was set to 3,000×, and the electron beam voltage was set
to 5 kV.

FIGs. 2A to 2G illustrate powder morphology images
of the examples and comparative examples. Referring to
10 Example 3 and Comparative Example 4, greater grain growth
was observed in Comparative Example 4 with a magnesia
content exceeding 70 vol%. Example 3 allowed coating,
but Comparative Example 4 did not allow coating.
Comparative Example 2 containing magnesia in an amount
15 below 10 vol% showed no great surface difference compared
with Comparative Example 1.

FIG. 3 shows EDS images of a granule according to
an example of the present disclosure.

For energy dispersion spectroscopy (EDS), JSM-
20 IT700HR model (JEOL) was used. The magnification was set
to 3,000×, and the electron beam voltage was set to 5 kV.
A small amount of granular powder was sampled and
attached onto a carbon tape, coated with gold, and
analyzed.

25 Referring to FIG. 3, the EDS images were for Example

3, and different types of materials (yttria powder and magnesia powder) were uniformly mixed on the granular powder.

Table 3 shows XRD data of the granular powders according to the examples and comparative examples of the present disclosure.

The X-ray diffraction (XRD) analysis was performed using Empyrean XRD instrument (Malvern Panalytical). Cu was used for an X-ray tube, and for X-ray generation, the voltage was set to 40 kV and the current was set to 30 mA. A theta-2theta scan was conducted over the 10° to 90° range. The fraction or reference intensity ratio (RIR) was measured by the RIR method. The fraction indicates the relative ratio of strongly appearing peaks. The d-value corresponds to the distance between lattice planes (Å) according to the peak position.

TABLE 3

Condition No.	XRD				
	Substance	Crystalline structure	Peak position (°)	d (Å)	Fraction (%)
Comparative Example 1	Y ₂ O ₃	Cubic	29.158	3.06025	100
Comparative Example 2	Y ₂ O ₃	Cubic	29.212	3.05470	100
Comparative Example 3	Y ₂ O ₃	Cubic	29.150	3.06131	93
	MgO	Cubic	42.871	2.10855	7
Example 1	Y ₂ O ₃	Cubic	29.152	3.06170	79
	MgO	Cubic	42.865	2.10789	21
Example 2	Y ₂ O ₃	Cubic	29.149	3.06111	58

	MgO	Cubic	42.866	2.10800	42
Example 3	Y ₂ O ₃	Cubic	29.153	3.06074	35
	MgO	Cubic	42.886	2.10700	65
Comparative Example 4	Y ₂ O ₃	Cubic	29.158	3.06025	12
	MgO	Cubic	42.909	2.10600	88

FIGs. 4A to 4G show graphs illustrating the XRD analysis results of the granular powders according to the examples and comparative examples of the present disclosure.

5 FIG. 4A shows the graph of Comparative Example 1, FIG. 4B of Comparative Example 2, FIG. 4C of Comparative Example 3, FIG. 4D of Example 1, FIG. 4E of Example 2, FIG. 4F of Example 3, and FIG. 4G of Comparative Example 4.

10 Compared with the conventional yttria powder (Comparative Example 1), Comparative Example 3 was observed to contain two types of powders (yttria and magnesia powders), which were not combined, distributed on the granule surface and, even after heat treatment,
15 to have 30 to 80% of Y₂O₃ cubic crystalline phase and 20 to 70% of MgO cubic phase rather than a mixed phase.

 The heat treatment was performed to remove organic additives introduced for granule manufacture and to provide flowability and strength for maintaining granule
20 shape and enabling coating.

 The granular powders of the examples and comparative examples were used to form coating layers by atmospheric

plasma spraying (APS). Particularly, Al 6061 substrates (20 mm × 20 mm × 3 mm) were used as base substrates. The plasma-spraying conditions for coating formation are shown in Table 4.

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TABLE 4

Classification	Voltage (V)	Current (A)	Power (kW)	Powder delivery rate (g/min)	Substrate distance (mm)
Comparative Example 1	60-80	400-700	35	10	130-180
Comparative Example 3				8.2	
Example 1				9.1	
Example 2				9.8	
Example 3				9.5	
Comparative Example 4				9.3	

Table 5 shows the PSA analysis results of the examples and comparative examples of the present disclosure, and FIGs. 5A to 5G show graphs illustrating the PSA analysis results. PSA was measured by wet analysis using LA-960 model (HORIBA). The refractive index values applied were 2.111-0.100i for Y₂O₃, 1.760-0.100i for MgO, and 1.333 for solvent DI water, and measurements were conducted in the transmittance range of 95 to 70% without using ultrasound waves and dispersants.

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FIG. 5A shows the graph of Comparative Example 1, FIG. 5B of Comparative Example 2, FIG. 5C of Comparative

Example 3, FIG. 5D of Example 1, FIG. 5E of Example 2, FIG. 5F of Example 3, and FIG. 5G of Comparative Example 4.

D10, D50, and D90 represent characteristic particle sizes in the cumulative volume distribution of particle sizes, wherein D10 indicates the particle size at the first 10% of the cumulative volume distribution, and D50, also called the median particle size, indicates the particle size at 50% of the cumulative volume distribution. D90 indicates the particle size at 90% of the cumulative volume distribution.

TABLE 5

Condition Number	Grain size (μm)	
Comparative Example 1	D10	23.5
	D50	33.0
	D90	46.2
Comparative Example 2	D10	21.8
	D50	33.1
	D90	48.4
Comparative Example 3	D10	22.4
	D50	32.5
	D90	46.1
Example 1	D10	23.0
	D50	34.6
	D90	49.8
Example 2	D10	23.0
	D50	36.5
	D90	54.8
Example 3	D10	28.7
	D50	41.1
	D90	61.8
Comparative Example 4	D10	27.8
	D50	42.6

	D90	66.13
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FIGs. 6A to 6F show SEM images of coating layer sections for the examples and comparative examples of the present disclosure.

With respect to SEM, the magnification was set to 5 300× and the electron beam voltage was set to 20 kV. The hardness (Hv) values of Comparative Examples 1, 2, and 3 and Examples 1, 2, and 3 were measured to be 450 Hv, 450 Hv, 600 Hv, 650 Hv, 780 Hv, and 700 Hv, respectively. Comparative Example 4 could not be measured for hardness
10 due to impossibility of coating.

Referring to FIGs. 6A to 6F, when the cross-sections of the coating layers were observed by SEM after atmospheric plasma spraying (APS), a clear phase separation between yttria and magnesia was confirmed
15 since yttria and magnesia are not mutually soluble in the solid state.

However, as a result of atmospheric plasma spraying (APS) coating, the grain size of the coating layer was 0.1 μm up to 3 μm under the conditions in Comparative
20 Examples 1 and 2, but the grain size was reduced to 0.1 μm up to 0.4 μm under the conditions in Example 2, indicating that the grain size can be controlled.

FIGs. 7A and 7B illustrate the grain size distributions for Comparative Example 1 and Example 2,

and FIGS. 8A and 8B illustrate crystalline structures for Comparative Example 1 and Example 2, and FIGS. 9A and 9B illustrate the crystallographic orientations for Comparative Example 1 and Example 2. Measurements were
5 conducted using electron backscatter diffraction (EBSD).

FIGS. 8A and 9A show the results for Comparative Example 1, and FIGS. 8B and 9B show the results for Example 2. Referring to FIGS. 7A and 7B, the average grain size of Comparative Example 1 was 2.64 μm , but the
10 average grain size of Example 2 was 0.37 μm , which was reduced compared with that of Comparative Example 1. Referring to FIGS. 8A and 8B, the crystalline structure of Example 2 was further micronized than that of Comparative Example 1. Referring to FIGS. 9A and 9B, the
15 crystallographic orientation of Example 2 was distributed more uniformly than that of Comparative Example 1.

The yttria (Y_2O_3)-magnesia (MgO) mixed powder for coating manufactured using a spray granulation process according to an embodiment of the present disclosure
20 exhibited a reduction in contaminant particles through micronization of the crystalline phase in the coating layer compared with the conventional yttria (Y_2O_3) powder. Furthermore, compared with the conventional yttria (Y_2O_3) coating layer, the mixing with magnesia (MgO) can enhance
25 the strength of the coating layer and lower the

manufacturing cost.

The use of yttria (Y_2O_3)-magnesia (MgO) mixed powder for coating can achieve an excellent plasma-resistant coating on a component with a complex shape, and the
5 formation of the plasma-resistant coating on a substrate with high impact resistance can achieve overcoming the limitations of sintered bodies. Furthermore, a coating layer with suppressed grain growth can lead to grains smaller than that in the conventional yttria coating
10 layer as well as reduced surface roughness. This can improve process stability and yield by generating smaller and fewer contaminant particles during plasma etching than conventional coatings to thereby reduce the adverse effects of such particles.

15 The specified matters and limited embodiments and drawings such as specific elements in the present invention have been disclosed for broader understanding of the present invention, but the present invention is not limited to the embodiments, and various modifications
20 and changes are possible by those skilled in the art without departing from an essential characteristic of the present invention. The spirit of the present disclosure is defined by the appended claims rather than by the described embodiments above, and all changes and
25 modifications that fall within metes and bounds of the

claims, or equivalents of such metes and bounds are
therefore intended to be embraced by the range of the
spirit of the present disclosure. Additionally, the
respective embodiments may be combined and operated
5 together as needed.

WHAT IS CLAIMED IS:

1. A method for manufacturing a composite powder,
the method comprising:

5 preparing a slurry in which a yttria powder, a
magnesia powder, a solvent, and an organic additive are
mixed;

performing ball milling on the slurry; and

drying the slurry to produce a granular powder
10 containing the yttria powder and the magnesia powder,
wherein the granular powder contains yttria crystals
and magnesia crystals.

2. The method of claim 1, wherein the slurry is
15 spray-dried to produce the granular powder.

3. The method of claim 1, wherein the organic
additive is contained in an amount of 1 to 6 wt% relative
to the total weight of the yttria powder and the magnesia
20 powder.

4. The method of claim 3, wherein the organic
additive contains a dispersant and a binder, and relative
to 100 wt% of the organic additive, the dispersant is
25 contained in an amount of 30 to 90 wt% and the binder is

contained in an amount of 10 to 70 wt%.

5. The method of claim 4, wherein the binder is at least one of PVA, PVB, PVP, and PEO.

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6. The method of claim 1, wherein the RIR of the yttria powder to the magnesia powder contained in the granular powder is 30:70 to 80:20.

10 7. The method of claim 1, wherein the RIR of the yttria powder to the magnesia powder contained in the granular powder is 35:65 to 79:21.

8. The method of claim 1, wherein relative to a
15 total of 100 wt% of the yttria powder, the magnesia powder, and the solvent, the sum of the yttria powder and the magnesia powder is 30 to 70 wt%.

9. The method of claim 1, wherein in the granular
20 powder, relative to a total of 100 vol% of the yttria powder and the magnesia powder, the volume ratio (vol%) of the yttria powder to the magnesia powder is 30:70 to 70:30.

25 10. A composite granular powder, comprising a

yttria powder and a magnesia powder, wherein the volume ratio (vol%) of the yttria powder to the magnesia powder is 30:70 to 70:30.

5 11. The composite granular powder of claim 10, wherein the granular powder is produced by spray-drying a slurry in which the yttria powder, the magnesia powder, a solvent, and an organic additive are mixed.

10 12. The composite granular powder of claim 10, wherein the RIR of the yttria powder to the magnesia powder contained in the granular powder is 30:70 to 80:20.

15 13. The composite granular powder of claim 10, wherein the RIR of the yttria powder to the magnesia powder contained in the granular powder is 35:65 to 79:21.