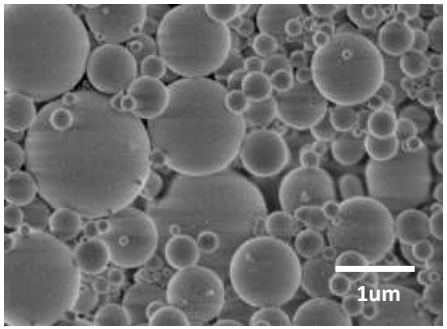
6/12/2016
Admatechs Co., Ltd
R&D Division

Admatechs Technical Overview

Admatechs Co., Ltd.

Contents

1. Company Profile



1-1. Admatechs Business Overview

1-2. Various size spherical silica (10nm – 30um)

1-3. Customize technology

- Surface treatment, Slurry, Classification, PSD control

2. Technical Data

2-1. Admanano Silica Master Batch

Surface treated Admanano silica can improve Composite physical properties.

2-2. Admanano Silica Powder

Admanano powder can improve powder hardener flowability and dispersibility.

Overview of Operation

Established February 1990

(History)

July	1994	New plant constructed (within TMC's Myochi Plant)
June	1996	National Commendation for Invention (awarded the Prime Minister Prize)
August	1999	Acquired ISO 9002 certification by the International Organization for Standardization (ISO)
October	1999	Established system with production capacity of 300 tons per month
May	2000	Construction of Technical Center (adjacent to TMC's biotechnology research institute)
October	2001	Relocation of Head Office (from Tokyo to the Technical Center)
October	2002	Acquired ISO 9001:2000 certification

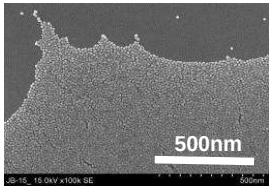
Capital 300 million yen : Toyota Motor Corporation 52.9%, Shin-Etsu Chemical Co., Ltd. 25.6%, Shin-Etsu Quartz Products Co., Ltd. 15.4%, Other 6.1%

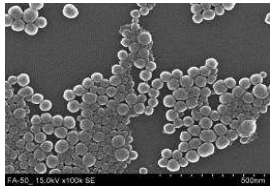
Business activities Manufacture and sale of oxide ceramic powders (silica, alumina, and composite oxides)

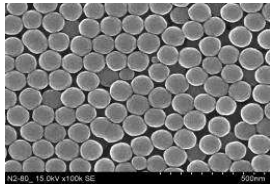
Location Aichi Prefecture, Japan

President Susumu Abe

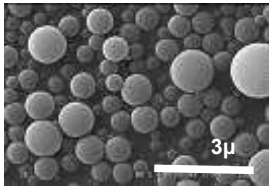
1-1. Admatechs Product Line Up

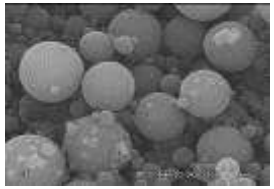


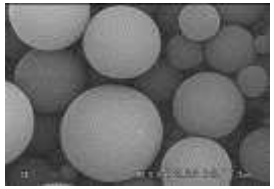




application







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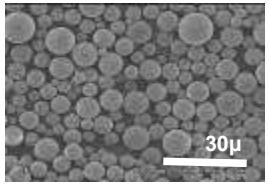
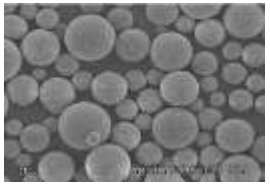
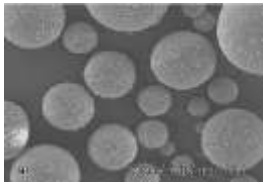






1 nm | 10 nm | 100 nm | 1000 nm (1μm) | 10μm | 100μm

Admanano

Nano sized silica
10 nm～100 nm
High Performance Nano Composite

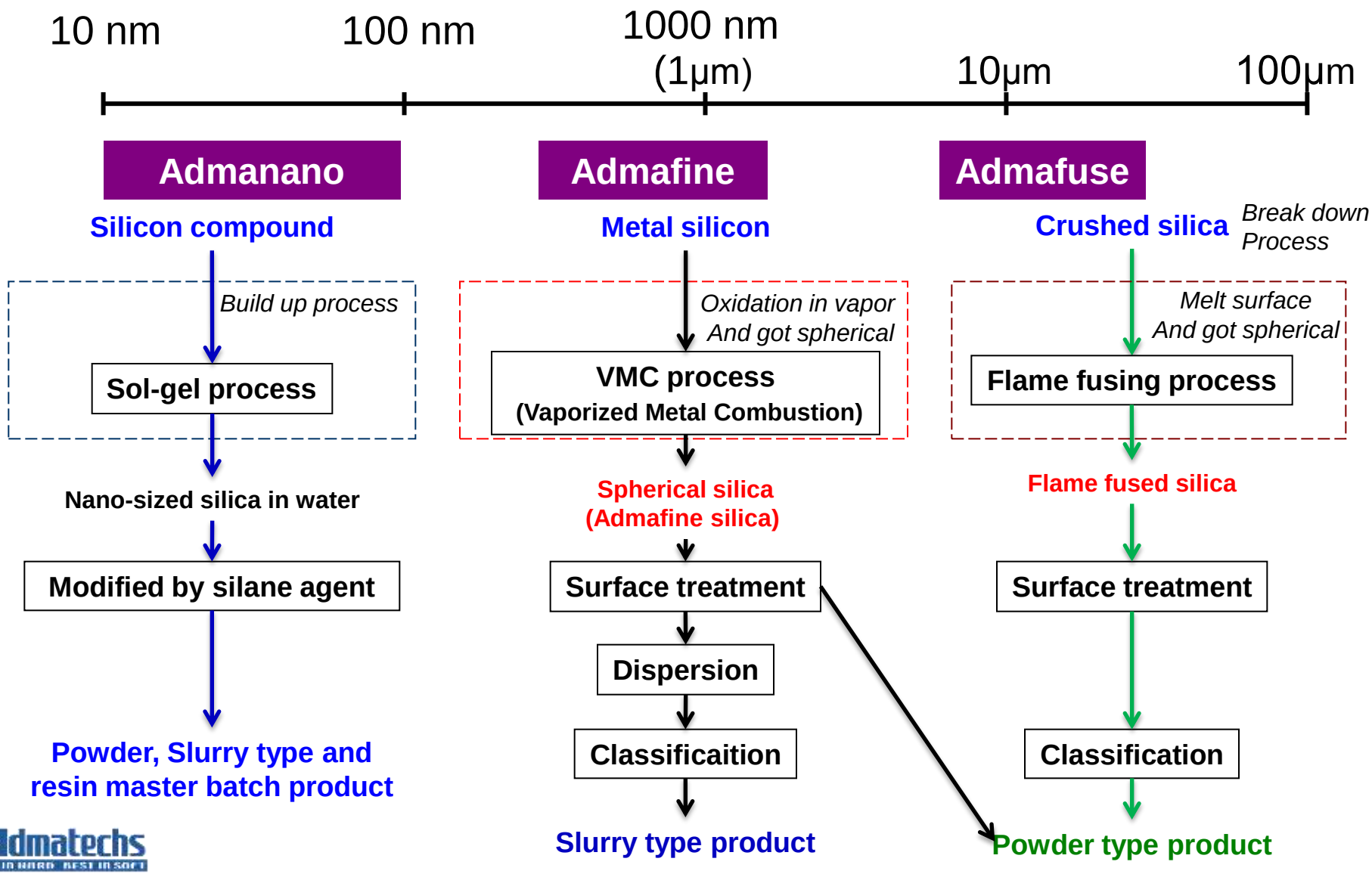
Admafine

VMC silica
0.2 μm～2.0 μm
Liquid encapsulation, sub-particles for EMC

Admafuse

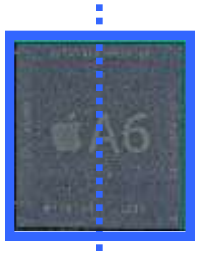
Flame fused spherical silica
5 μm～30μm
Main particles for EMC

1-1. Process Flow of Admatechs Products

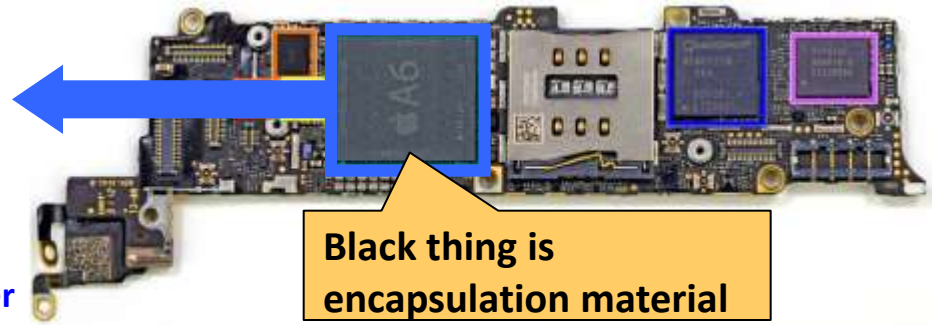


1-1. Application Example of Admatechs Products

Electronic Device

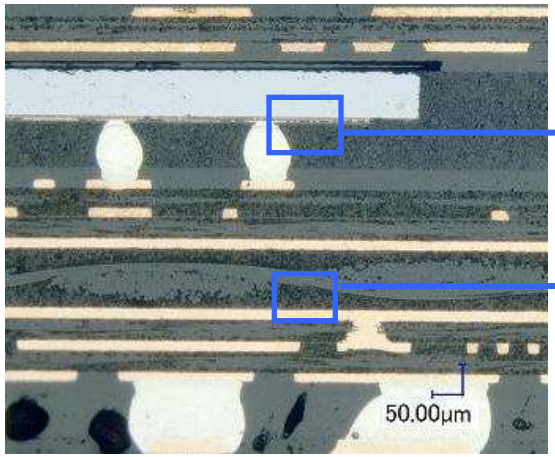


A6 : Application Processer
Cross Section



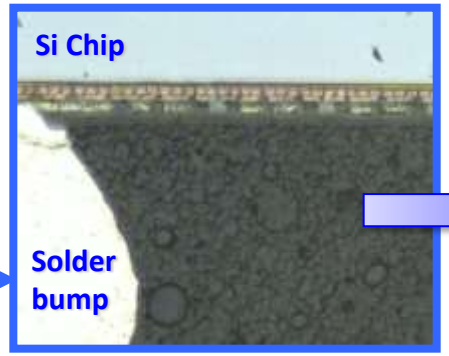
Black thing is encapsulation material

iPhone

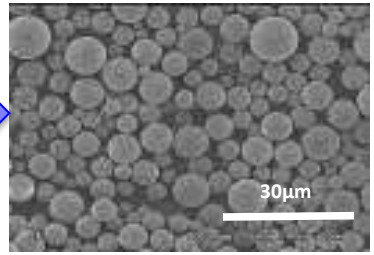


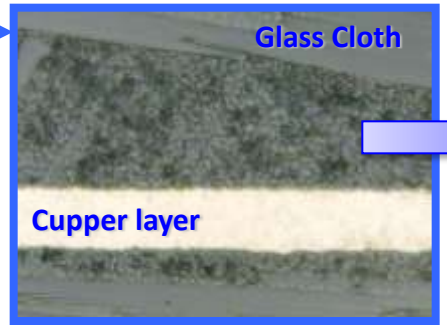
Mold
Under Fill

Substrate
layer

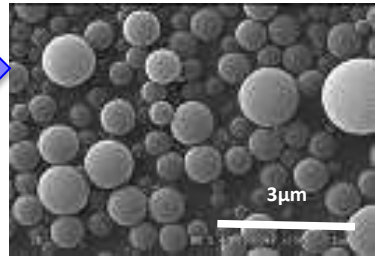


Admafuse : 5um silica





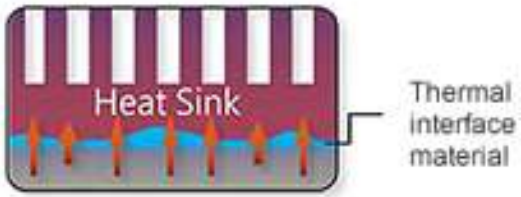
Admafine : 0.5um silica



1-1. Application Example of Admatechs Products

Thermal Interface Material

Schematic representation of two surfaces in contact and heat flow across the interface.

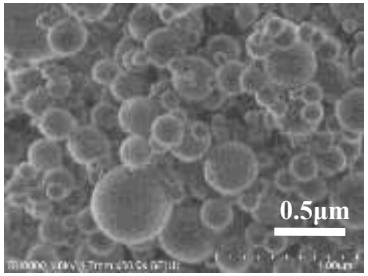


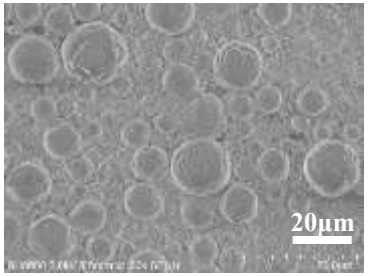
Interface material compressed between two contacting surfaces.

Thermally Conductive Sheet

Thermally Conductive Grease

Admafine Alumina : 0.6um, 10um

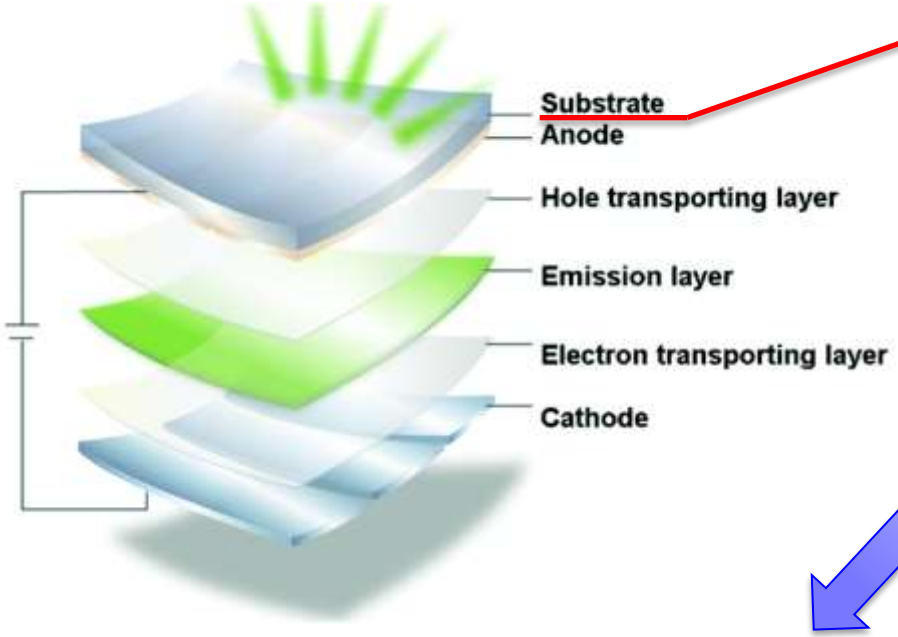




- **High Thermal Conductivity.**
- **Low Viscosity, High Flexibility.**

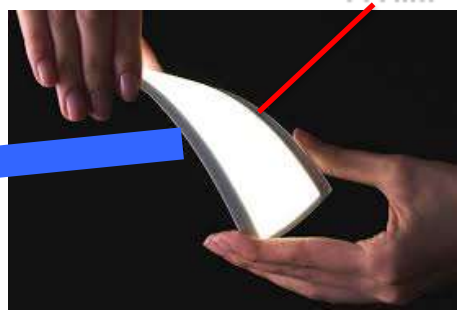
1-1. Application Example of Admatechs Products

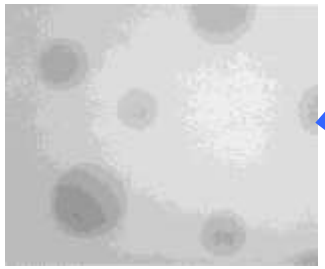
OLED



Glass Substrate (Traditional Rigid Substrate)

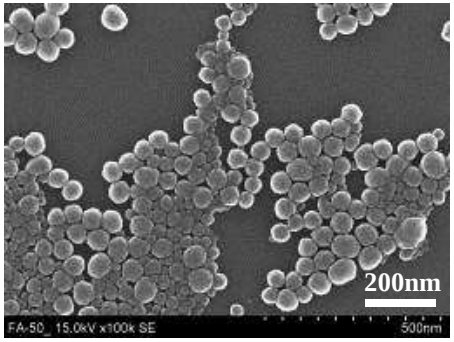
PI Film (Flexible Transparency Substrate)





PI Film, Cross Section Image

Admanano : 50nm silica

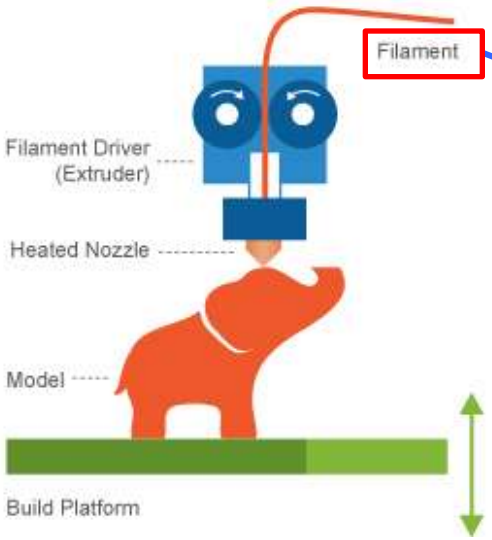


- Reduce CTE, improve physical properties
- High Transparency due to nano size and no agglomeration.

1-1. Application Example of Admatechs Products

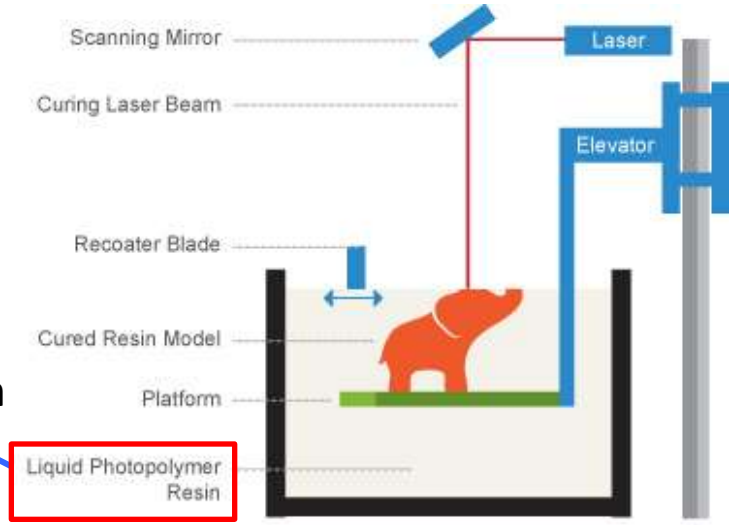
3D Printer

Fused Deposition Modeling (FDM)



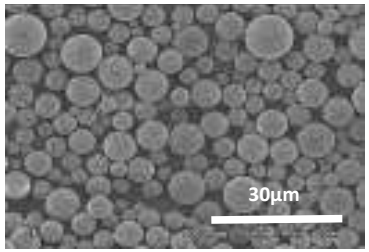
ABS, PLA,
Other resin

Stereo lithography (SLA)

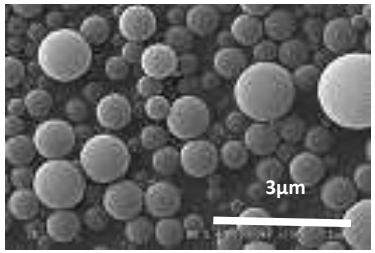


Acryl,
Other Resin

Admafuse : 5um silica

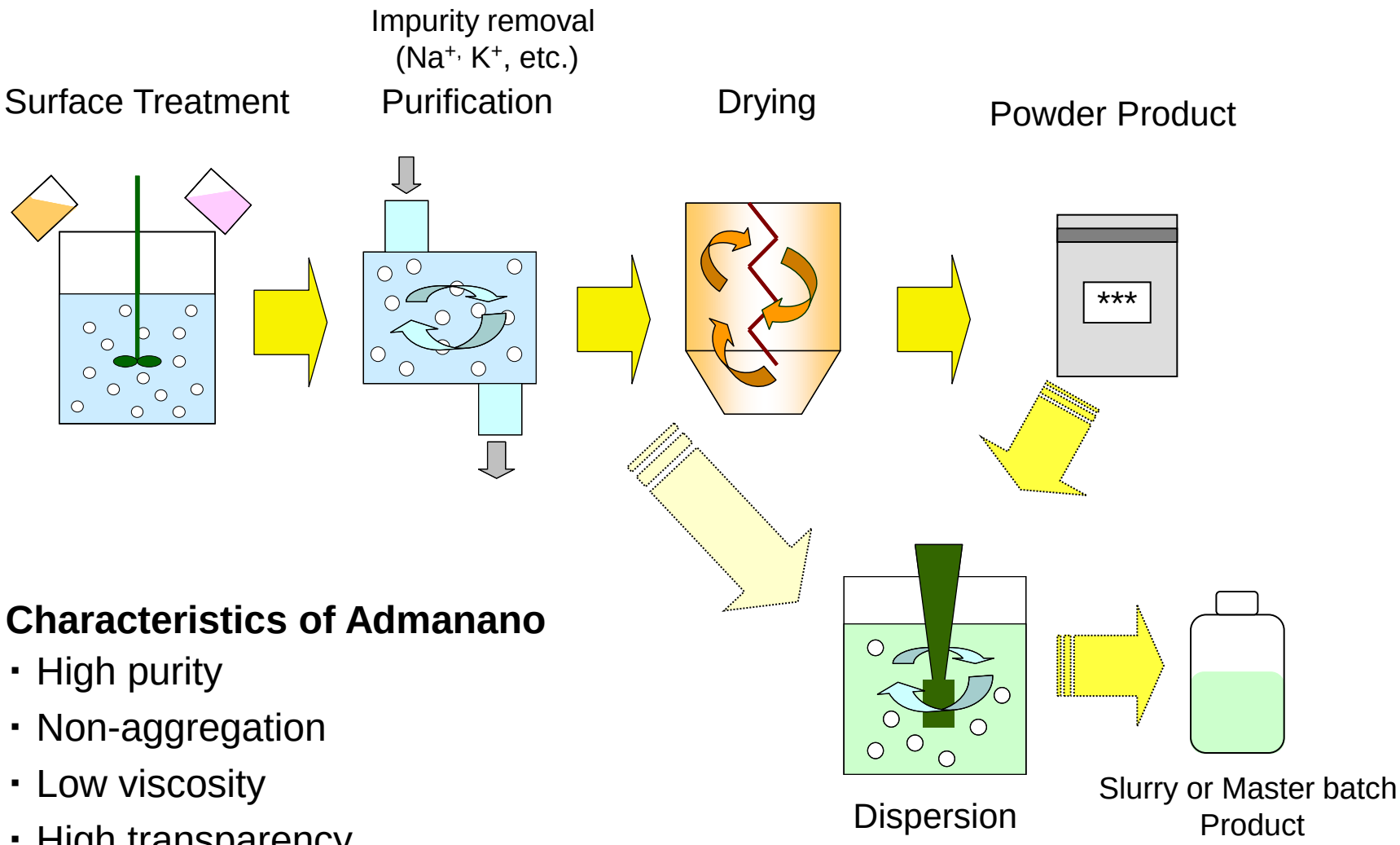


Admafine : 0.5um silica



- **Low Viscosity.**
- **Reduce CTE,**
  Improve physical properties.

Admanano Manufacturing Process (10 – 100nm)



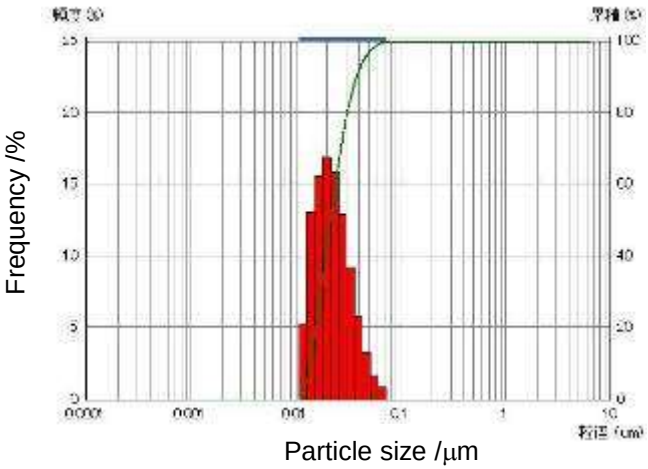
Characteristics of Admanano

- High purity
- Non-aggregation
- Low viscosity
- High transparency

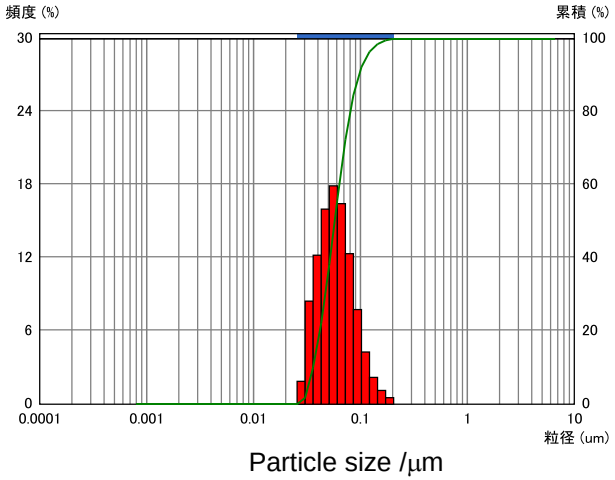
1-2. Various Size Spherical Silica

Admanano Silica Line Up

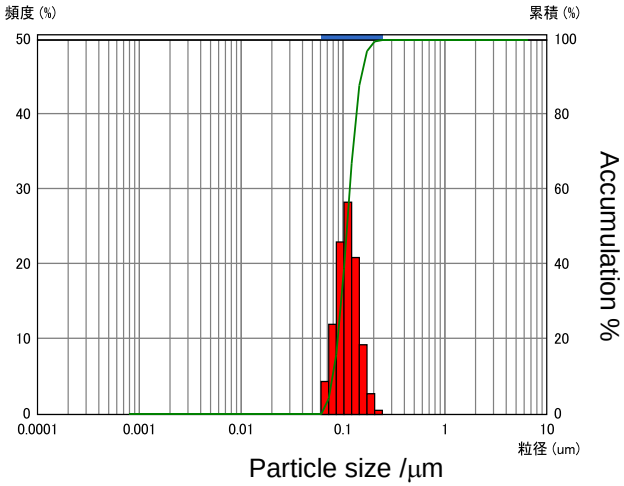
10nm

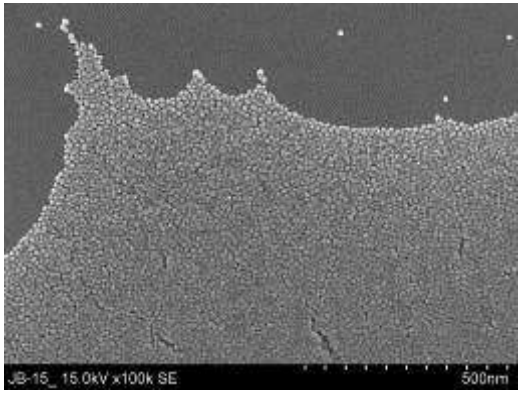


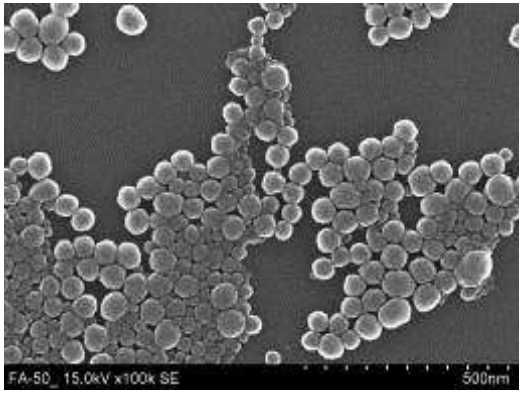
50nm

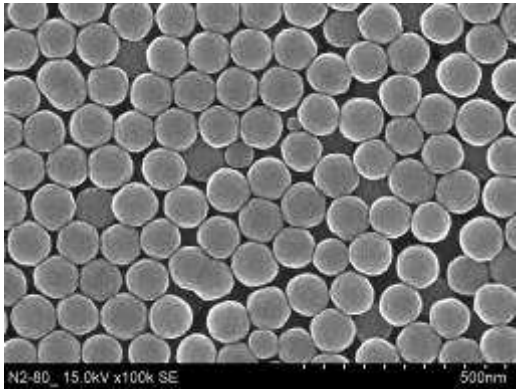


100nm



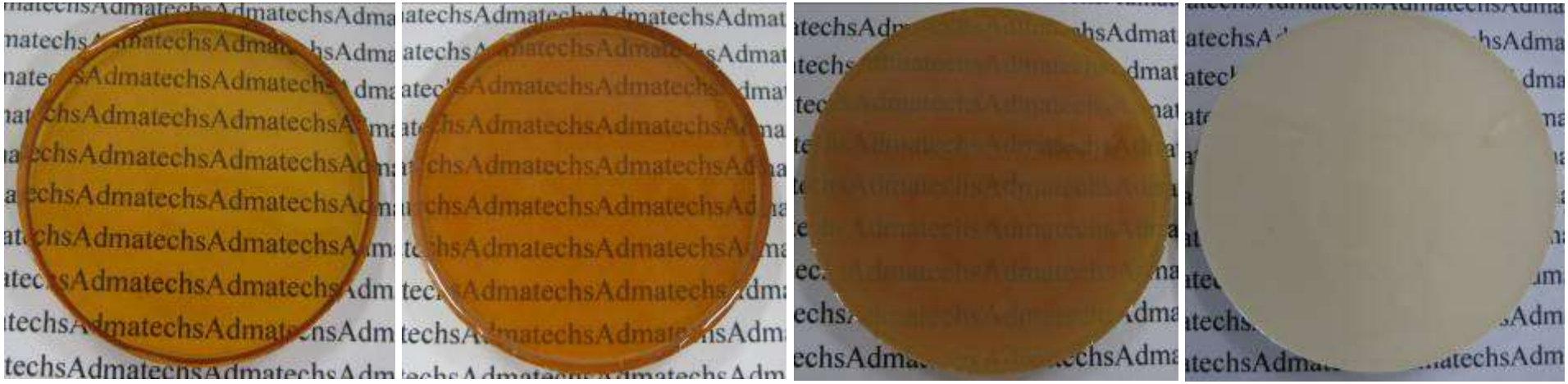






Admanano Silica Optical Property

10nm 15nm 50nm 300nm



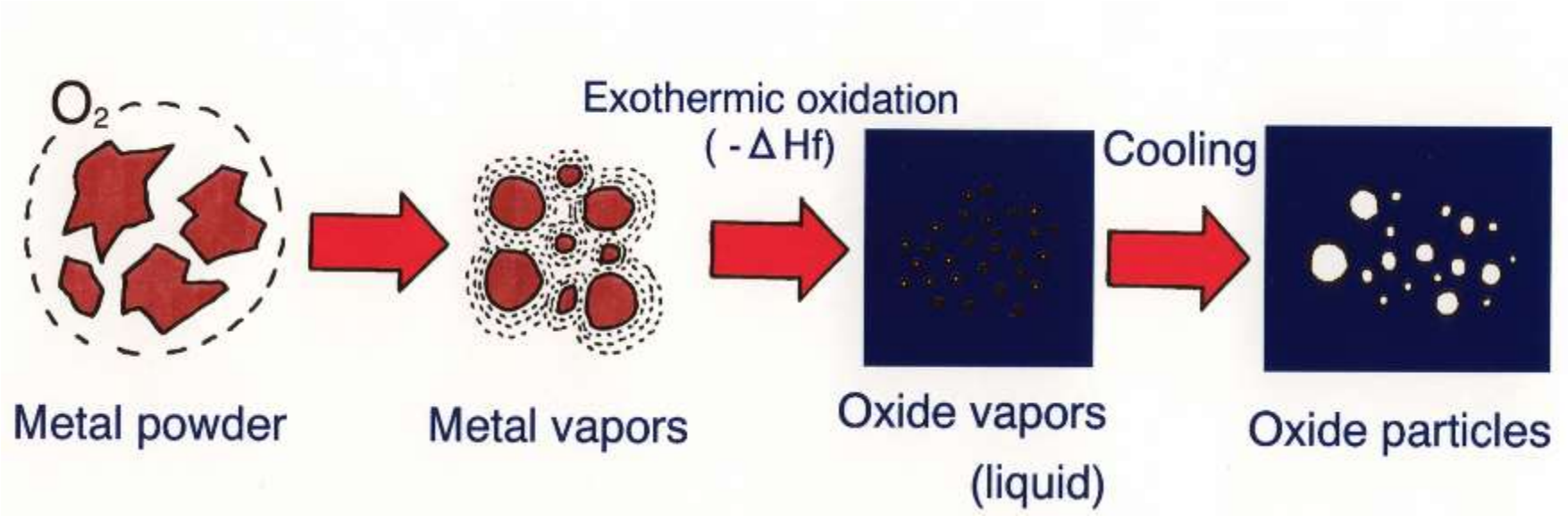
Thickness: ca.5mm

fine ← Particle Size → coarse

High ← Transparency → Low

Admafine Manufacturing Process (0.2 – 2um)

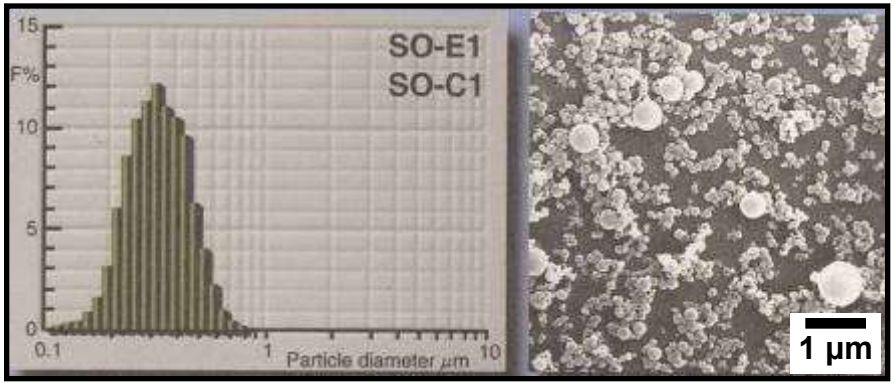
Core technology of ADMATECHS is Vaporized Metal Combustion (VMC) method. The VMC method provides fine spherical silica and alumina particles from metal powder by direct oxidation.



1-2. Various Size Spherical Silica

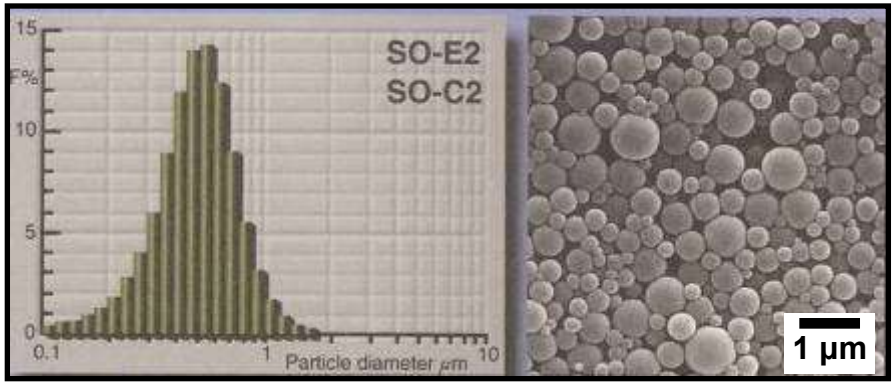
Admafine Product Line Up

Particles size distribution and SEM Images



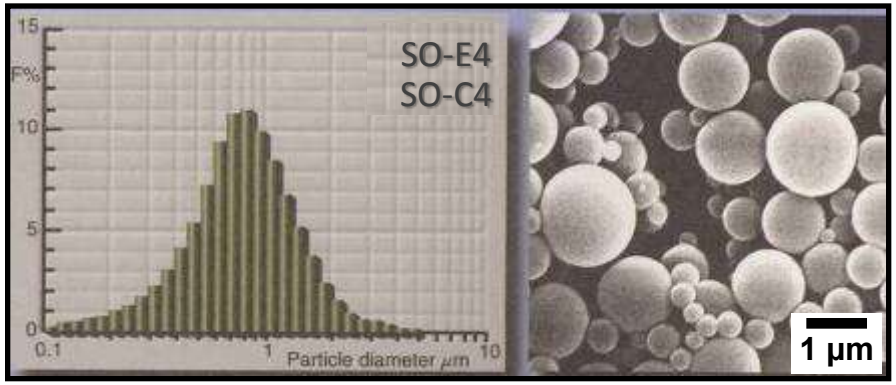
Particle diameter: 0.3 μm

Specific surface area: 10 ~ 20 m^2/g



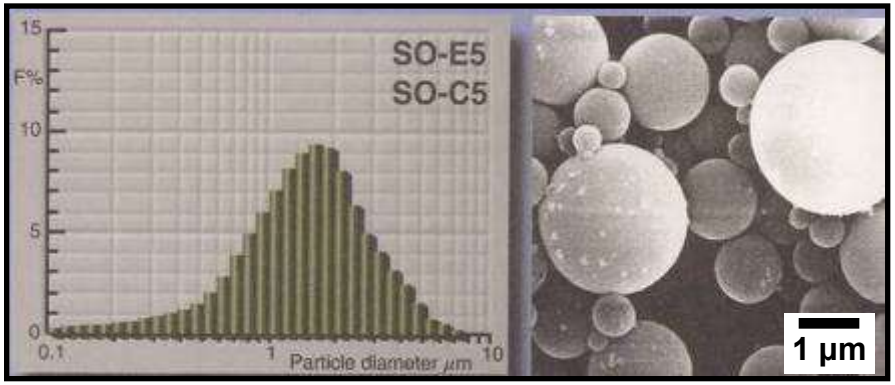
Particle diameter: 0.5 μm

Specific surface area: 5 ~ 9 m^2/g



Particle diameter: 1 μm

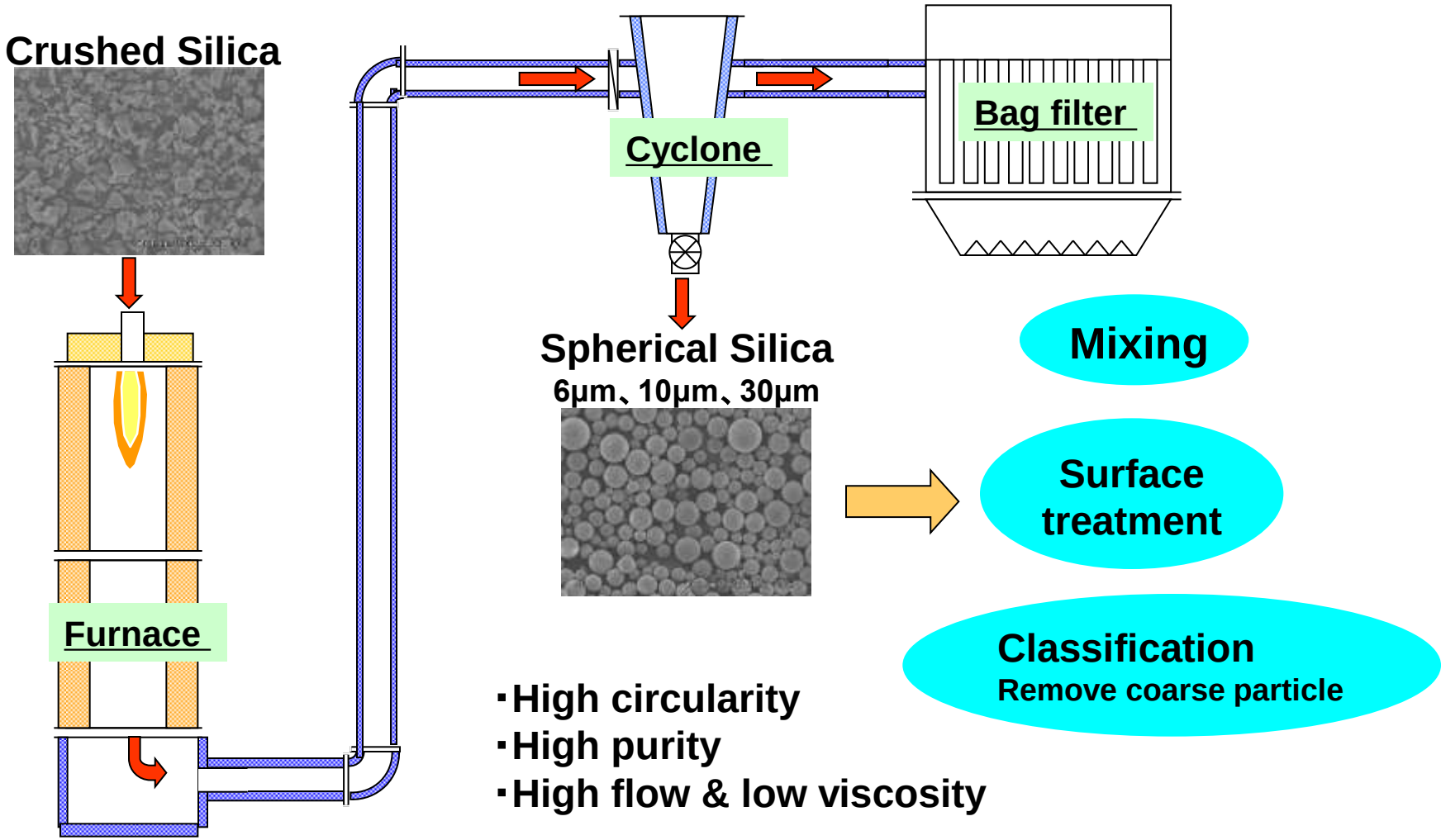
Specific surface area: 3 ~ 6 m^2/g



Particle diameter: 1.5 μm

Specific surface area: 2 ~ 5 m^2/g

Admafuse Manufacturing Process (5 – 30um)

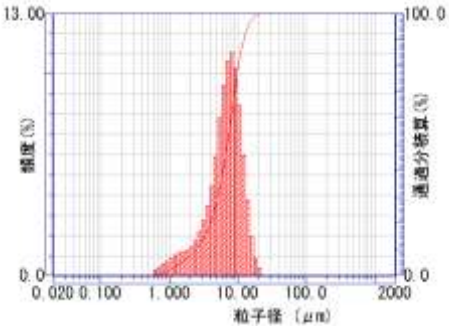
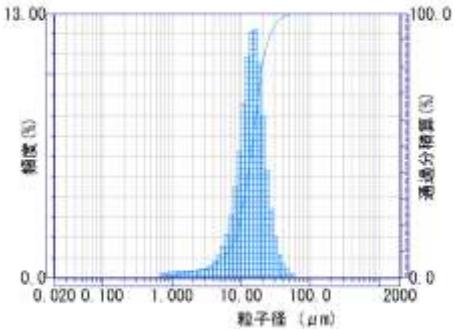
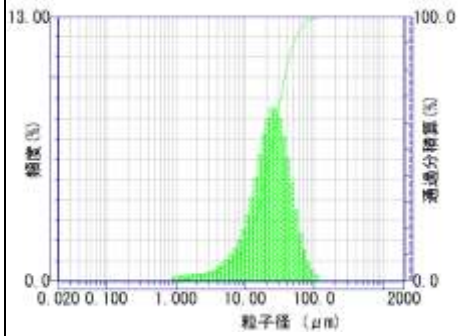
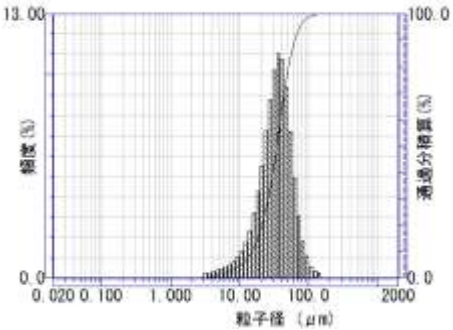
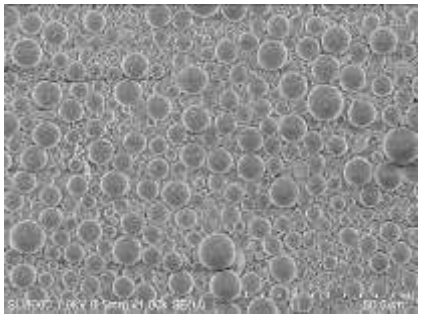
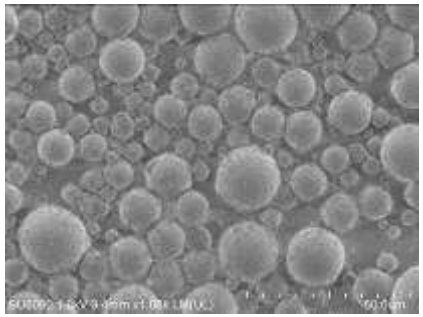
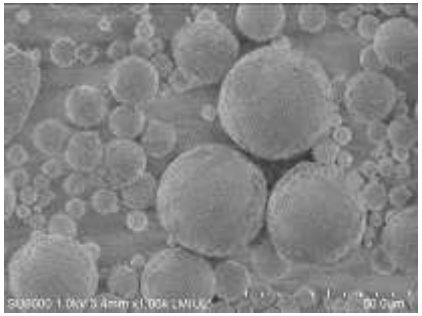
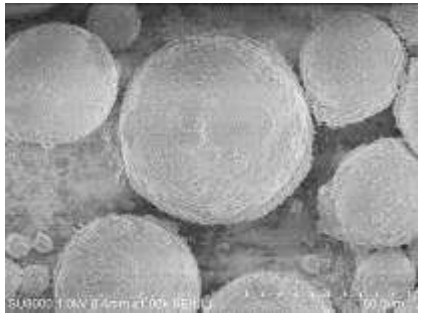


- High circularity
- High purity
- High flow & low viscosity

1-2. Various Size Spherical Silica

Admafuse Product Line Up

Product name

FE9-	FEB-	FED-	FEF-
			
6μm	10μm	20μm	30μm
			

- Surface Treatment Technology, “SQ Treatment”.

<SQ Treatment Concept>

Characteristics

1) Low surface reactivity toward epoxy group
2) Low agglomeration
3) Low adhesion to wall of apparatus
4) Easy to disperse into resin

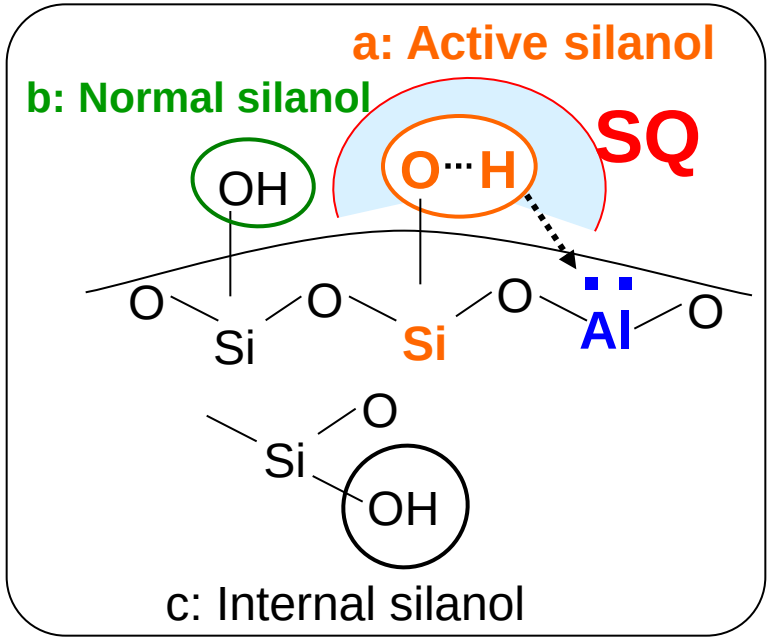
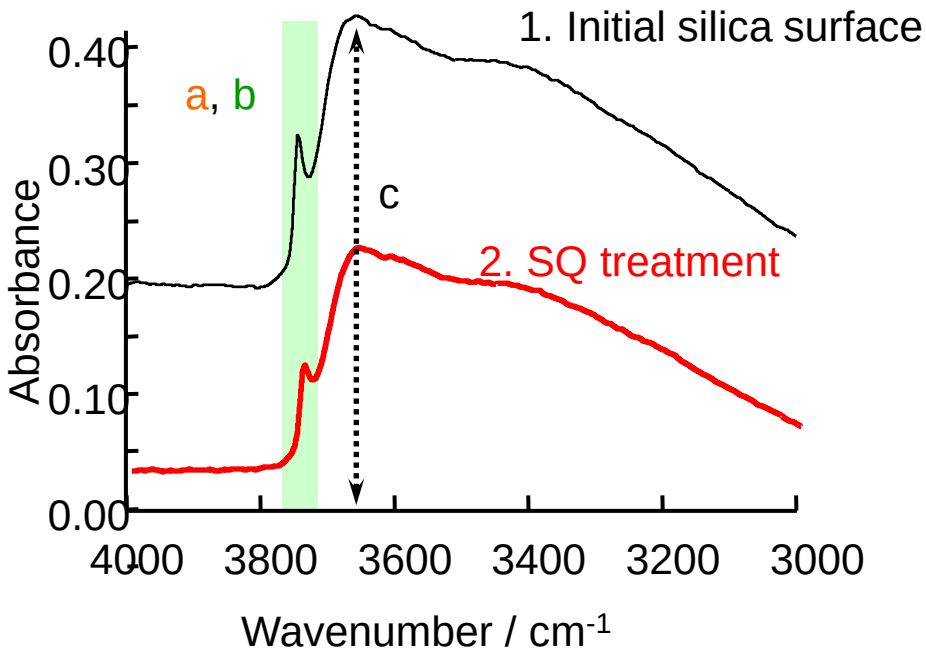


Fig. FT-IR spectra



1-3. Customize Technology
- Surface Treatment Technology, "Layer Concept".

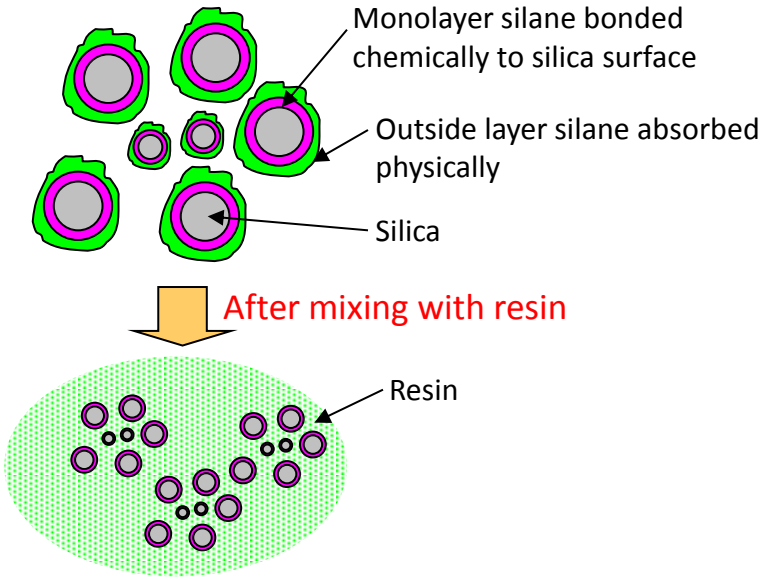
Silica surface modified with silane coupling agent are beneficial for;

(1) Low resin viscosity

(2) Less agglomeration

(3) Better compatibility with epoxy resin

Admatechs Surface Treated Silica

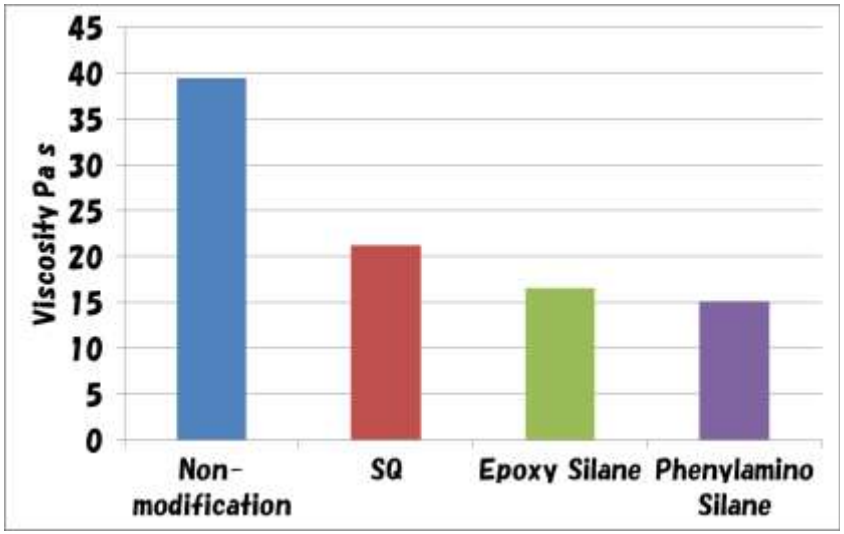


Viscosity Data

Resin : Bis F + Bis A epoxy resin

Silica : 0.5µm silica

Loading : 65wt%



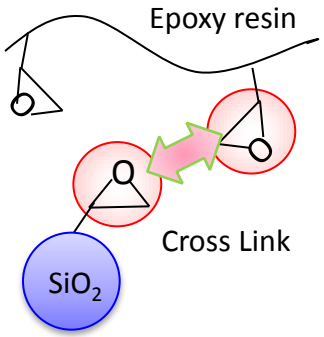
Share rate at 1 (1/sec)

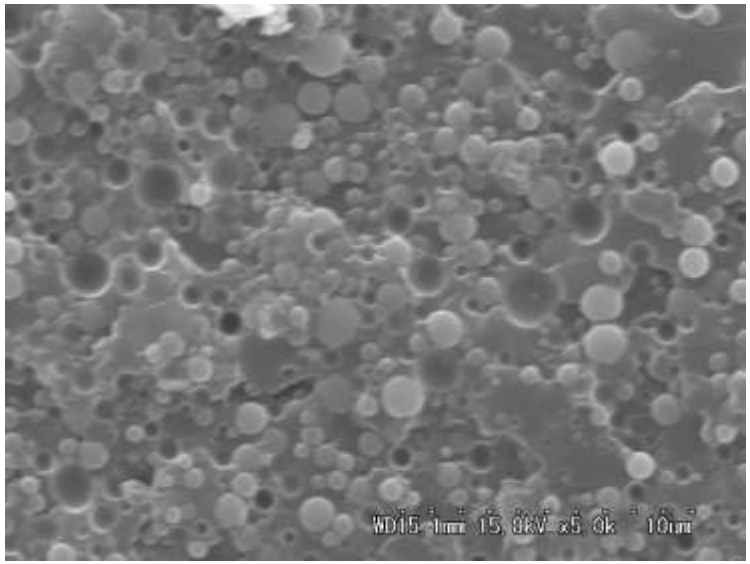
Surface treatment improves the compatibility with resin and reduces the viscosity.

Compatibility of Resin Matrix

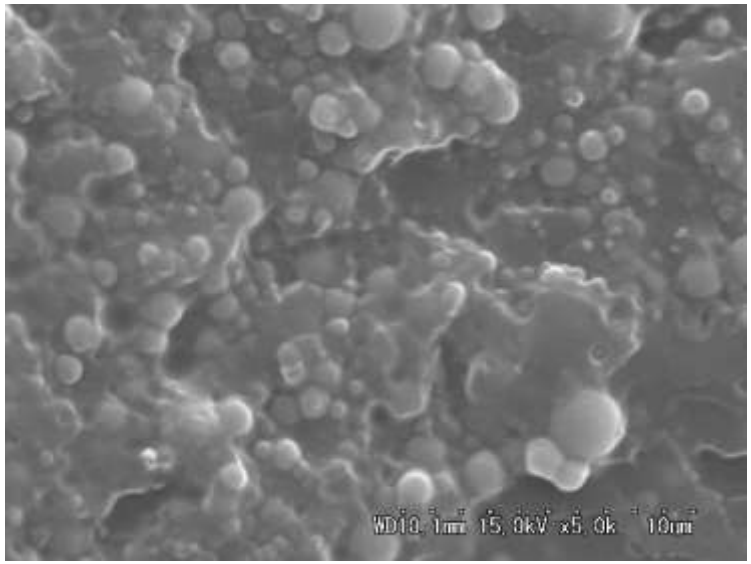
Test Condition

- Silica : 0.5µm silica
- Resin : Bis F + Bis A epoxy resin + Hardener : Imidazole
- Evaluation : Observation of fracture surface after curing by SEM





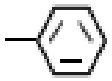
Non-Surface Treated Silica



Surface Treated Silica, (phenyl amino, etc…)

Surface treatment improves the adhesion between silica surface and resin.
This result improves physical properties of compounds.

Example of Surface Treatment Agents

Chemical Structure	Name	Chemical Structure	Name
$(CH_3O)_2Si(CH_3)_2$	Dimethyl silane	$(CH_3O)_2Si(CH_3)C_3H_6OCH_2CH(O)CH_2$	Epoxy silane
$(CH_3O)_3Si(CH_2)_5CH_3$	Hexyl silane	$(CH_3O)_3SiC_3H_6NH$ 	Phenyl amino Silane
$(CH_3O)_3SiC_6H_5$	Phenyl silane	$(CH_3O)_3SiC_3H_6OC(=O)C(CH_3)=CH_2$	Methacryl silane
$(CH_3O)_3SiCH=CH_2$	Vinyl silane	$(C_2H_5O)_3SiC_3H_6N=C=O$	Isocyanate silane

Various surface treatment agents are available.

Slurry Production Technology (For Admafine)

Japan patent #3920582 and more patents under application screening.

Admafine slurry production scheme

Silica production technology

↓

Surface modification technology

↓

Dispersion technology

↓

Classification technology

↓

Admafine slurry (filler for substrate)

Agglomeration control
High silica loading (>70wt%)

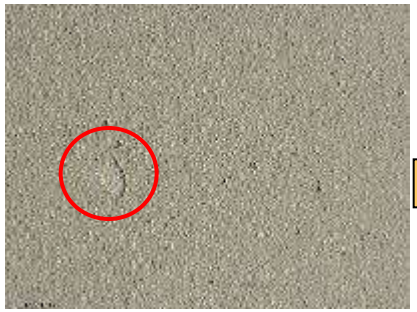


Photo1.
Bad dispersion slurry

Photo2.
Good dispersion slurry

Filtration by screening (5um)

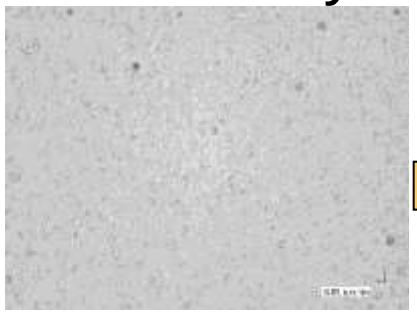


Photo3. Slurry containing Large particles

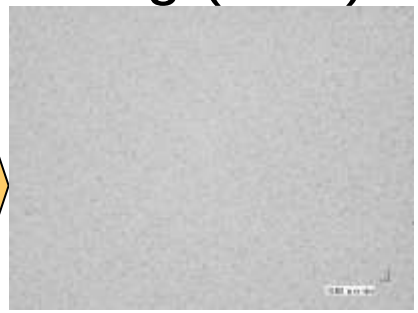
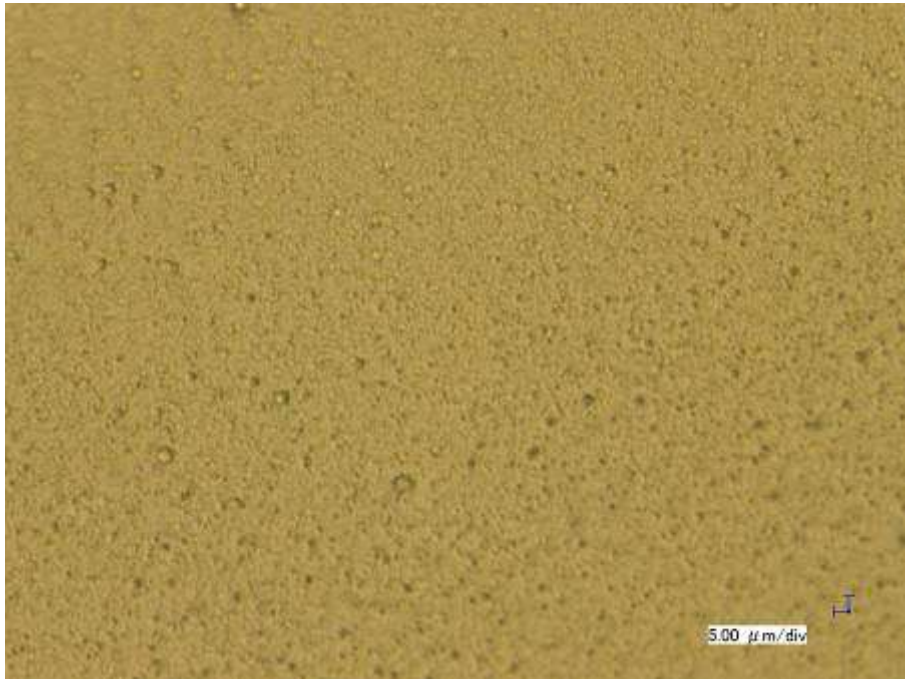
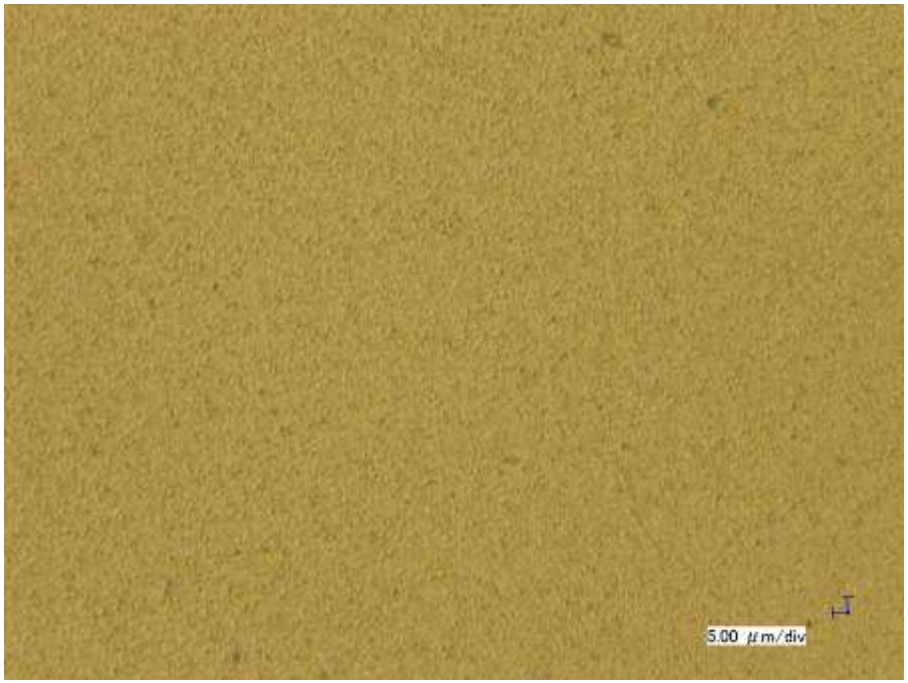


Photo4. No large particles found in slurry

New Classification Technology (for Admafine)



at 5μm
（containing 100ppm > 5μm particle）



at 3μm
（containing 10ppm > 3μm）

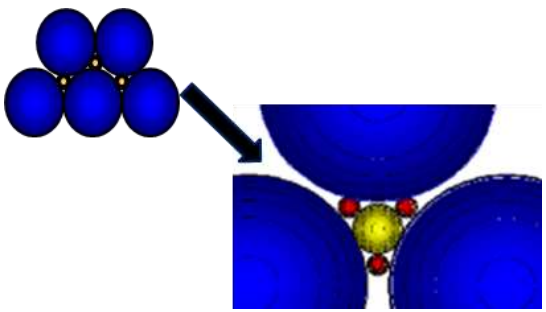
Photographs of 0.3μm particle size silica slurries（×1000）

Control of Particle Size Distribution

<Closest Packing Concept>

1) Combination of bigger particle and smaller particle

2) Submicron size & Spherical shape



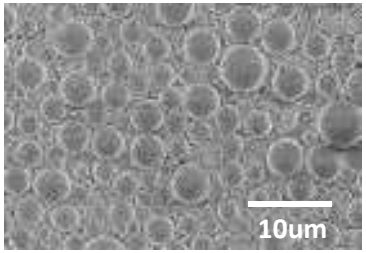
Resin Viscosity

Resin : Liquid Epoxy resin （BisA ＆ BisF mixed）

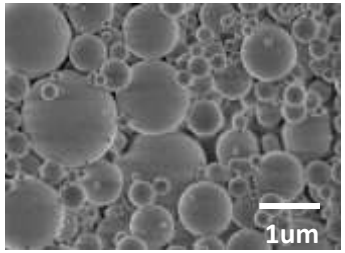
Filler : 5um silica (FE975A)

: 0.5um silica (SC200G-SQV)

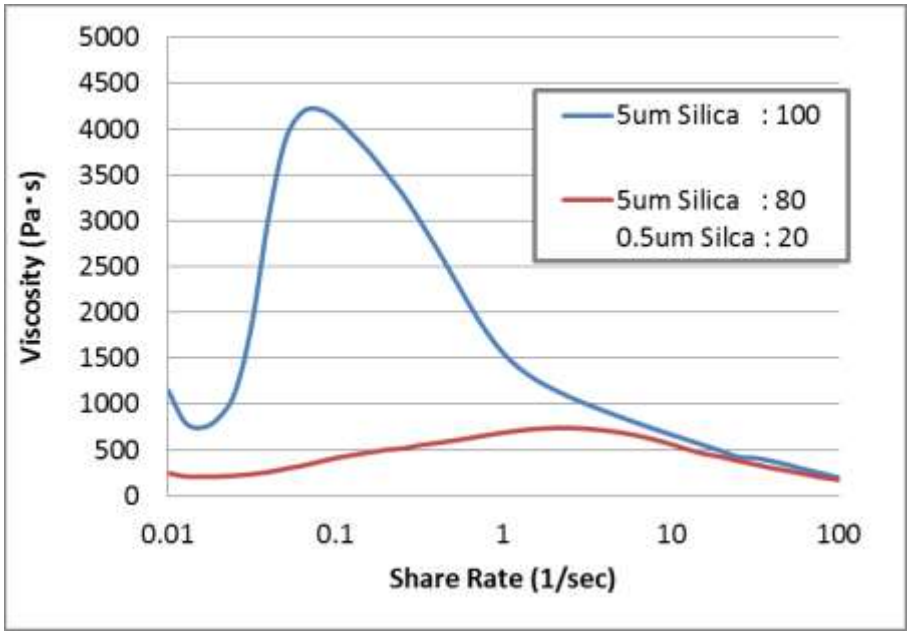
Loading : 80wt%



5um silica (FE975A)



0.5um silica (SC200G-SQV)



Closest Packing can improve the compound viscosity.

Control of Particle Size Distribution

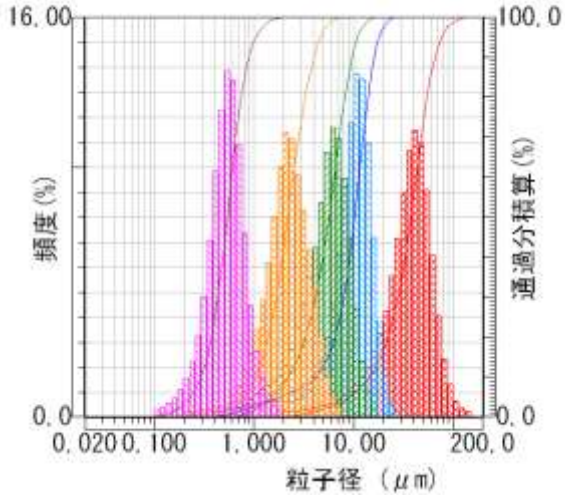
The product can be developed according to customer's demand by Admatechs.

Fused silica (2～30 μm)
Admafine silica (0.2～2 μm)

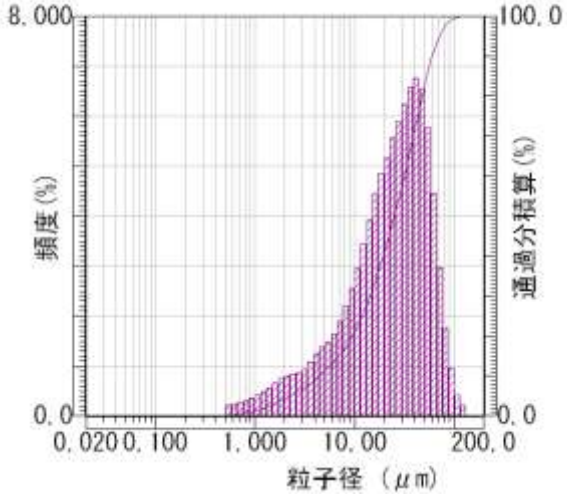
Customized silica

Input: customer's information

- **Particle diameter**
- **SSA**
- **Silica loading**
- **Classification**
  Remove coarse particle
- **Surface treatment**
  (Resin information)



Base silica



Customized silica

Contents

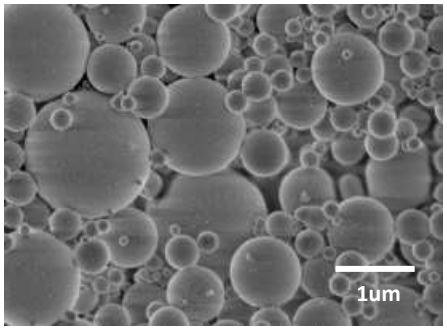
1. Company Profile

1-1. Admatechs Business Overview

1-2. Various size spherical silica (10nm – 30um)

1-3. Customize technology

- Surface treatment, Slurry, Classification, PSD control



2. Technical Data

2-1. Admanano Silica Master Batch

Surface treated Admanano silica can improve CFRP physical properties.

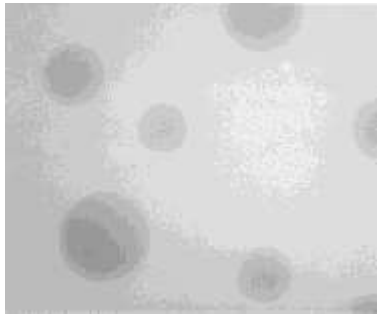
2-2. Admanano Silica Powder

Admanano powder can improve powder hardener flowability and dispersibility.

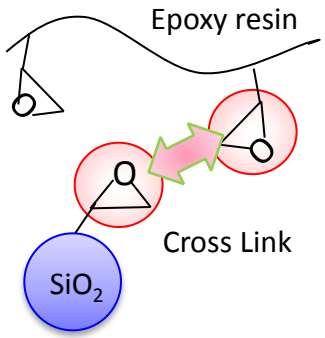
Effect of Admanano for Resin – Physical Properties

●Features of Admanano Master Batch

- **Uniformly surface treated spherical nano silica.**
- **Admanano silica can be dispersed in various resin system uniformly.**
- **Surface treated silica can react with resin system.**



Admanano in epoxy resin



Admatechs epoxy treated silica

Physical Properties

Resin : Bis F epoxy Resin
Admanano : 10nm epoxy treated silica
Loading : 15wt%
Hardener : Aromatic diamine
Curing Condition : 170℃, 2 hrs

Items	Flexural Strength (MPa)	Young's modulus (GPa)	Flexural Strength (MPa)	Young's modulus (GPa)
Temperature	20℃	20℃	140℃	140℃
Neat epoxy resin	88	3.1	20	0.5
Addition of Admanano	141	4.5	31	1.1

Each physical property was improved using Admanano Master Batch

2-1. Admanano Silica Master Batch -

Effect of Admanano for Resin – Fracture Toughness, CTE

Fracture Toughness

Resin : Bis F epoxy resin mixture
Micro Silica : 0.5um epoxy treated silica
Admanano : 50nm epoxy treated silica
Loading : 40wt%
Hardener : Aromatic diamine
Curing Condition : 170℃, 2 hrs.

Items	Fracture Toughness K_{IC} (@25℃) MPa·$m^{0.5}$
Micro Silica 40wt%	2.38
Micro Silica 30wt% Admanano 10wt%	2.61

Fracture Toughness was also improved using Admanano.

CTE Properties

Resin : Bis A + Bis F epoxy Resin
Silica : 1.5um, 0.5um, 50nm
Epoxy treated silica
Loading : 40wt%
Hardener : Aromatic diamine
Curing Condition : 170℃, 2 hrs

CTE	Control Resin	Silica Diameter		
		1.5µm	0.5µm	50nm
CTE1 (0-50C)	64	44	45	43
CTE2 (175-195C)	187	135	132	133
Tg	152	152	154	154

50nm silica can also reduce CTE as well.

Admatechs
BEST IN NANO BEST IN SOFT

2-1. Admanano Silica Master Batch -
Effect of Admanano for CFRP – Physical & CTE Data

1st Evaluation of CFRP using surface treated Admanano Silca

CF : PAN-3K (Plain fabrics 200g/m^2)

CF Loading : 60vol%

Resin : **Bis A epoxy Resin**

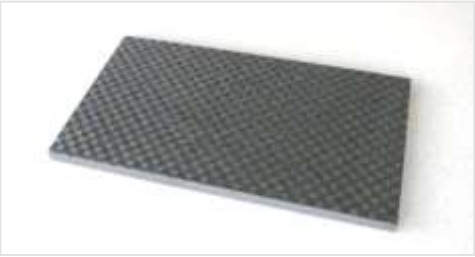
Admanano : **50nm epoxy treated silica (YA050A-JFL)**

Silica Loading : 15wt% for resin

Number of CF layer : 20 layers

Hardener : DICY

Curing Condition : Press molding 1Mpa, 130℃, 60 min



	Flexure Strength (MPa)	Flexure Modulus (GPa)	Tensile Strength (MPa)	Tensile Modulus (GPa)	ILSS (MPa)
CFRP	999	65	804	68	75
CFRP + Admanano	1135	69	971	79	80

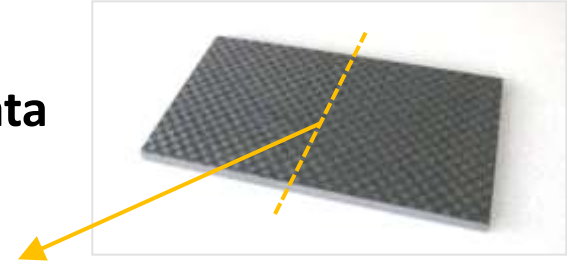
Z axis CTE	CTE1 (0-50℃) (ppm/K)	CTE2 (175-195℃) (ppm/K)
CFRP	39	211
CFRP + Admanano	35	182

In addition to reducing CTE. Admanano can improve CFRP strength, modulus and ILSS by 5~20%

Admatechs
BEST IN HARD BEST IN SOFT

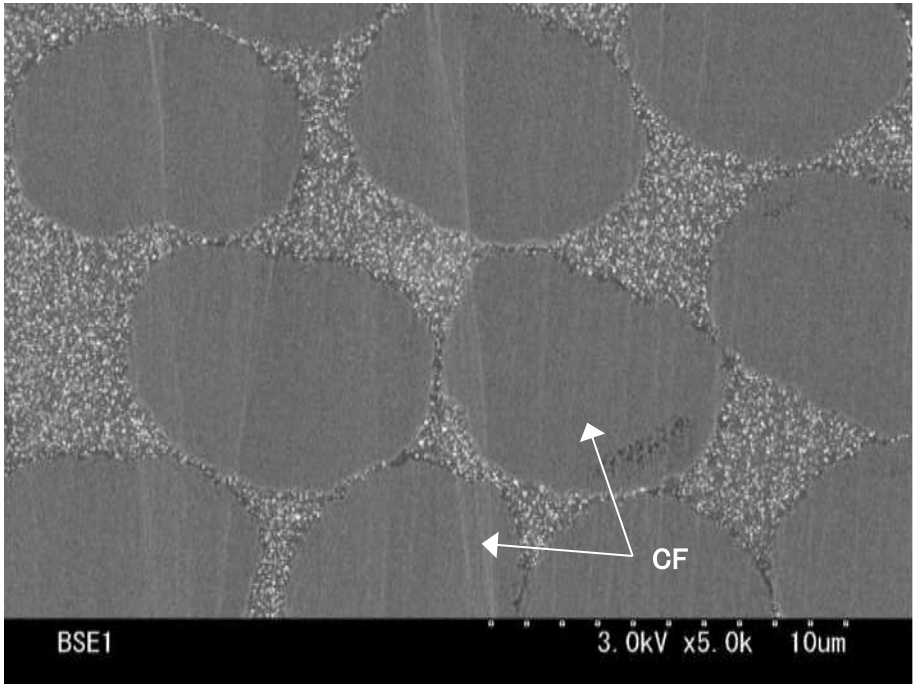
2-1. Admanano Silica Master Batch -
Effect of Admanano for CFRP – SEM Image

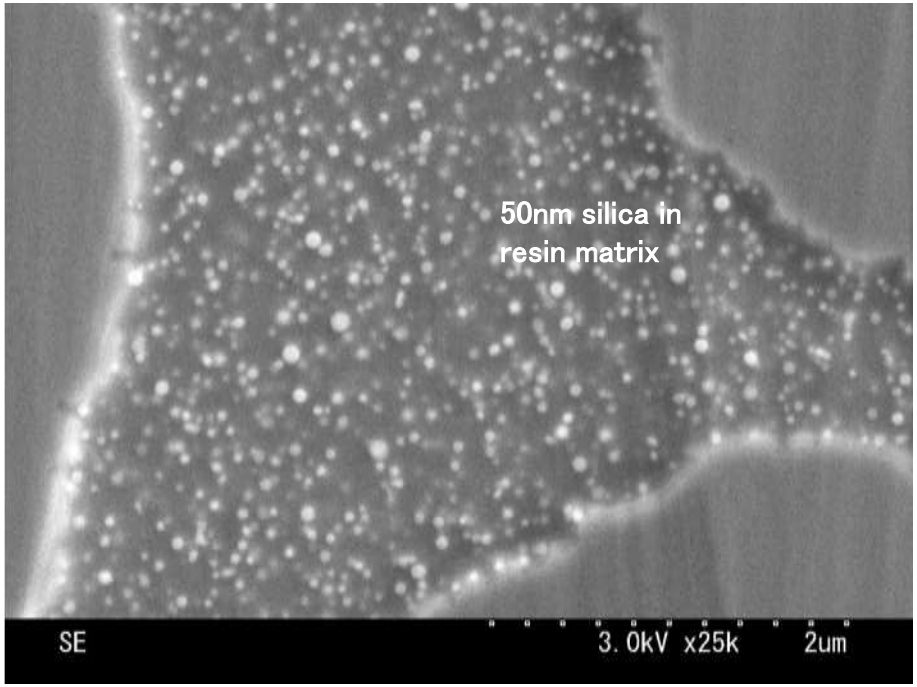
FE-SEM
CFRP Cross Section Data



Admanano : 50nm epoxy treated silica

Silica Loading : 15wt% for resin





50nm Admanano silica is dispersed inside CFRP uniformly.

Effect of Admanano for CFRP – Physical Data

2nd Evaluation of CFRP using surface treated Admanano Silca

CF : T700 12K (Toray)

CF Loading : 55vol%

Resin : **Bis A epoxy Resin**

Admanano : **50nm epoxy + more functional treated silica (YA050A-JFZ)**

Silica Loading : 15wt% for resin

Number of CF layer

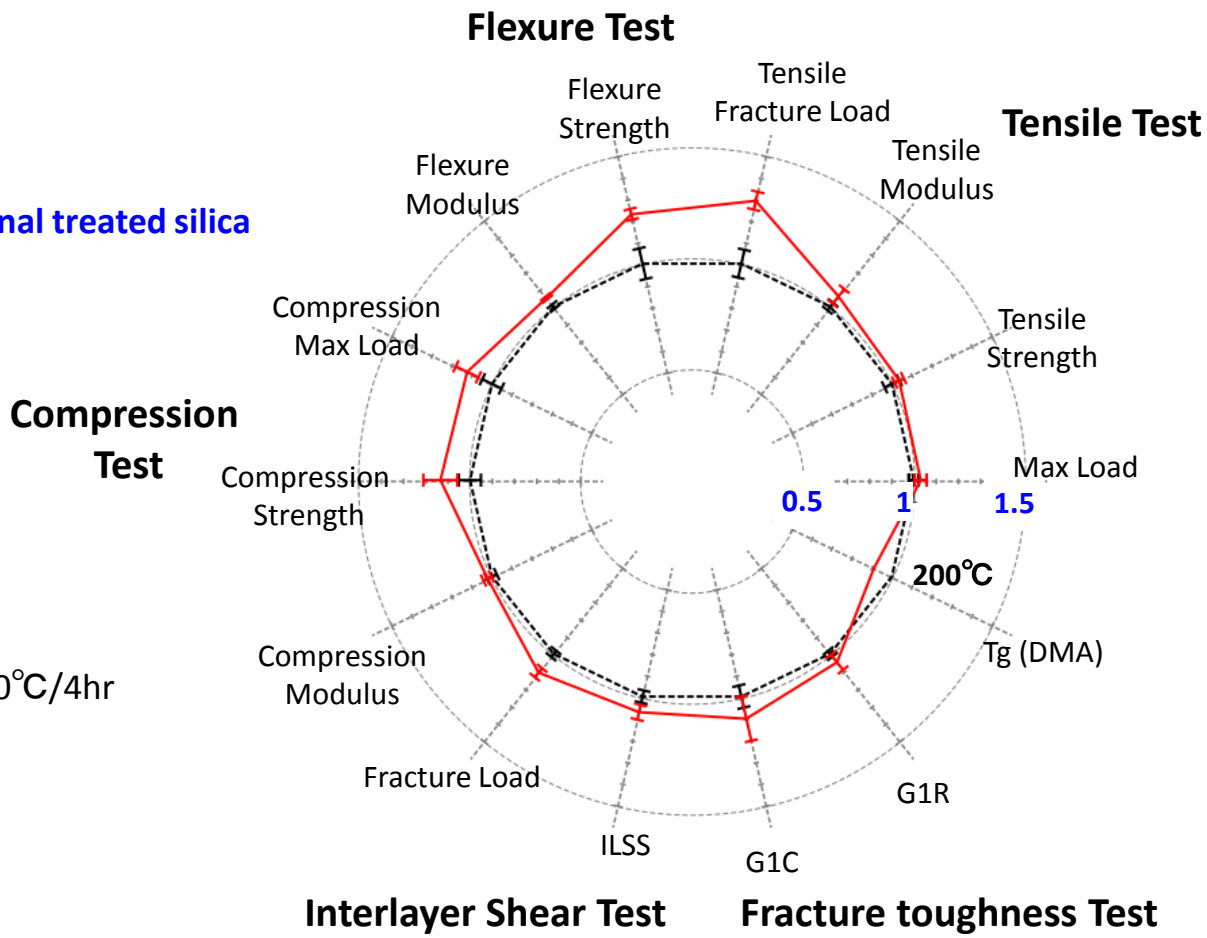
: 8 layers (Except ILSS)

: 14 layers for ILSS

Hardener : DDS (0.9 for resin)

Curing Condition : Press molding

0.3Mpa, 100℃/2hr, 180℃/4hr



Every physical property was improved by 5~20%.
Tg was decreased from 200℃ to 180℃ in this case.

2-1. Admanano Silica Master Batch -
Effect of Admanano for CFRP – Physical Data

2nd Evaluation of CFRP using surface treated Admanano Silca

CF : T700 12K (Toray)

CF Loading : 55vol%

Resin : **TGDDM**

Admanano : **50nm epoxy + more functional treated silica (YA050A-JFZ)**

Silica Loading : 15wt% for resin

Number of CF layer

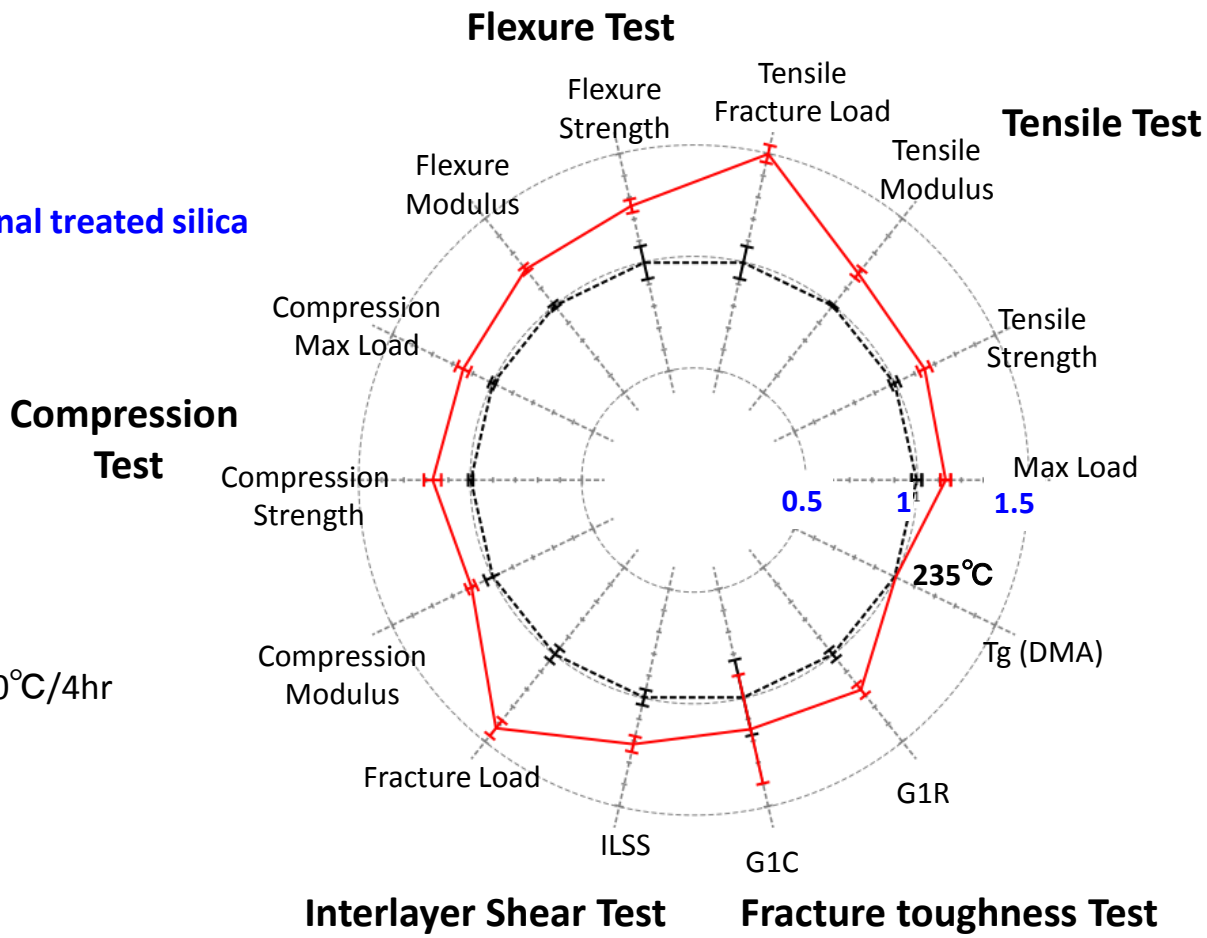
: 8 layers (Except ILSS)

: 14 layers for ILSS

Hardener : DDS (0.8 for resin)

Curing Condition : Press molding

0.3Mpa, 100℃/2hr, 180℃/4hr



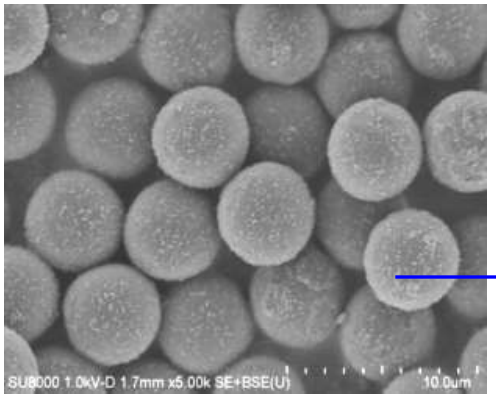
Every physical property was improved by 15~26%.
Tg was no change from base resin in this case.

Admatechs
BEST IN HARD BEST IN SOFT

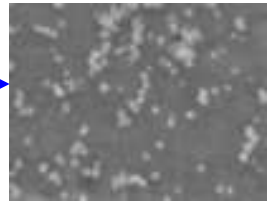
2-2. Admanano Silica Powder-
"Admanano" vs "Fumed Silica"

Dispersibility on Powder Surface

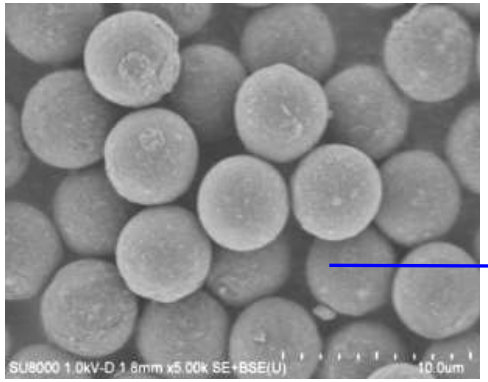
"Admanano" onto PS beads (0.5%)



No aggregation

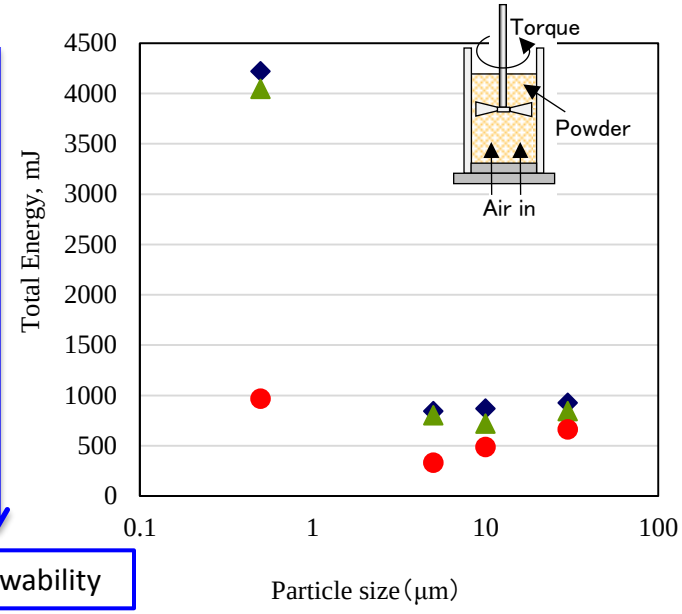


"Fumed Silica" onto PS beads (0.5%)



Aggregation about 0.5µm

Powder Flowability (Effect of Diameter)



High Flowability

◆ : **No addition**

● : **External addition of Admanano (0.3%)**

▲ : **External addition of fumed silica (0.3%)**

Admanano can improve the powder flowability compared to fumed silica.

2-2. Admanano Silica Powder-
Dispersibility in Organic Solvent

Admanano Powder

+ Solvent

Shaking by hands

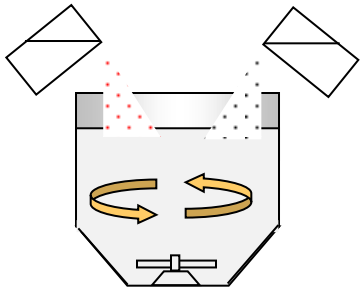
Admanano powder can be dispersed in organic solvent without any dispersers

Ex. Average particle size: 10nm
Surface treatment: Methacryl silane
Solvent: MEK
Silica loading: 50wt%

2-2. Admanano Silica Powder-

Powder Flowability Improvement

Dry mixing

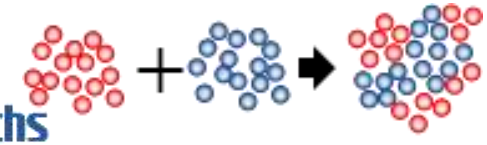


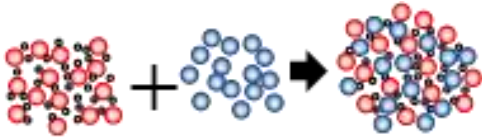
Without Admanano

Spherical silica (0.5um)
+
Pigment (0.05~0.2um) 3%

With Admanano

Spherical silica (0.5um) + Admanano 0.3%
+
Pigment (0.05~0.2um) 3%





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Effect of Admanano for Powder Hardener (DICY)

Powder Flowability

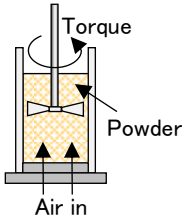
Hardener : DICY
3um diameter

Admanano : YA010C-SP3 (10nm)
1wt% for DICY

Mixing Method : Shaking by Hand

Sample	Flowability Energy (mJ) ※
DICY	400
DICY＋Admanano	100

※Smaller value means high flowability.



Admanano improves the powder flowability of DICY Significantly.

Dispersibility into Resin

Resin : Liquid Epoxy Resin

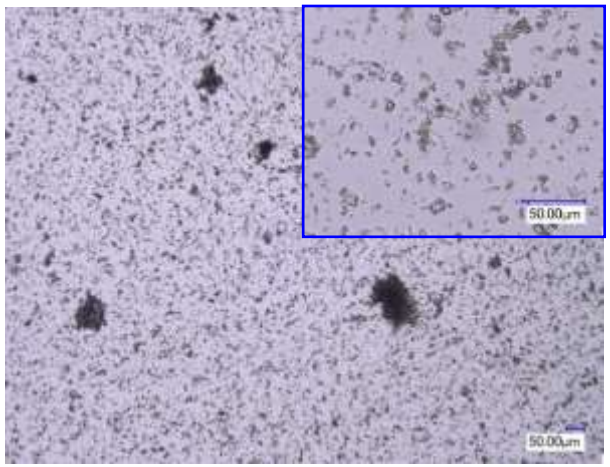
Hardener : DICY

Admanano : YA010C-SP3 (10nm)
1wt% for DICY

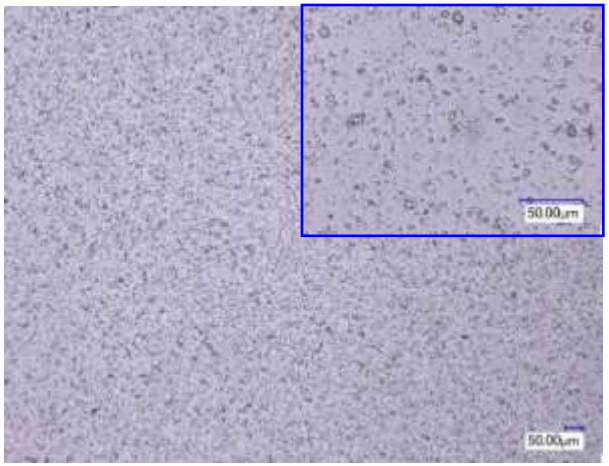
Loading : 5wt%

Mixing Method : Planetary Mill
2000rpm, 2min

Only DICY



DICY + Admanano



Admanano can improve the dispersibility of DICY Significantly.

2-2. Admanano Silica Powder-
Effect of Admanano for Powder Hardener (DDS)

Efficiency of Screening

Hardener : DDS, 4um diameter

Admanano : YA010C-SP3 (10nm)

1wt% for DDS

Mixing Method : Shaking by Hand

Vibrating Sieve Machine : 425um mesh

Humidification : 40 ℃, 80%RH, 12 days

Pressurization : 5kg, 1day

Evaluation Method : DDS 1g was placed on a sieve, then screened for 3min. After Sieving, the weight of residue was measured.

	Normal	Humidification	Pressurization
Only DDS			
Sieve Efficiency(%)	27	12	4
DDS + Admanano			
Sieve Efficiency(%)	97	90	53

DDS got agglomeration because of aging or their own weight, then it became harder to sieve.

On the other hand, Admanano could avoid agglomeration, so the sieve efficiency was improved.

- Summary -

- **Various Size Silica Fillers and Customize Technology**

  Admatechs can supply various customized silica filler based on demand.

- **Admanano Silica Master Batch**

  Surface treated Admanano silica can improve CFRP physical properties.

- **Admanano Silica Powder**

  Admanano can improve powder flowability and dispersibility.

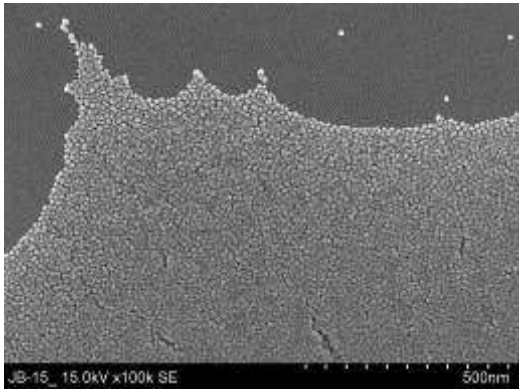
Thank you!

Admanano Product Line Up

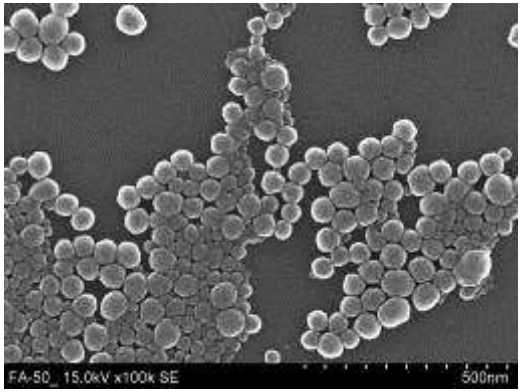
	Powder					Slurry		Master Batch	
Purity	Particle Size (nm)	SSA (m2/g)	Surface treament			Solvent	SiO_2 Contents	Resin	SiO_2 Contents
			Phenyl	Methacryl	Vinyl				
High purity type (Standard)	10	300	YA010C-SP3	YA010C-SM1	YA010C-SV1	MEK MIBK PGM Cyclohexanone , etc.	20-60 wt%	Epoxy resin Acryl resin , etc.	20-60 wt%
	50	60	YA050C-SP3	YA050C-SM1	YA050C-SV2				
	100	30	YC100C-SP3	YC100C-SM1	YC100C-SV2				

※NOTE: This table shows "available products" including grades which are under development.

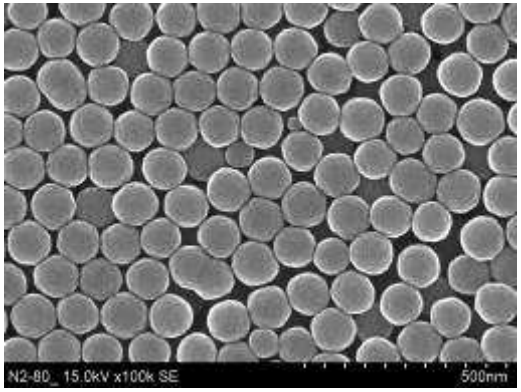
10nm



50nm



100nm



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Admanano Epoxy Resin Master Batch Product Line Up

Product Name※1	Diameter※2	Surface treatment	Silica Loading	Epoxy Resin Type	EEW
YA010A-JEW	10nm	Epoxy	20wt%	Bis Phenol F (High Purity Type)	230g/mol
YA050A-JEW	50nm		50wt%		370g/mol
YA010A-JGY	10nm	Epoxy	25wt%	Bis Phenol F (Standard Type)	270g/mol
YA050A-JGY	50nm		50wt%		440g/mol
YA010A-JFL	10nm	Epoxy	20wt%	Bis Phenol A	260g/mol
YA050A-JFL	50nm		40wt%		360g/mol
YA010A-JFZ	10nm	Epoxy + More Functional Treatment	20wt%	Bis Phenol A	270g/mol
YA050A-JFZ	50nm		40wt%		360g/mol
YA010A-JFS	10nm	Epoxy	20wt%	Alicyclic	180g/mol

※1 These products line up contain under development ones.

※2 Average diameter was calculated from SSA.

Admatechs can try various resin master batch based on customer request.

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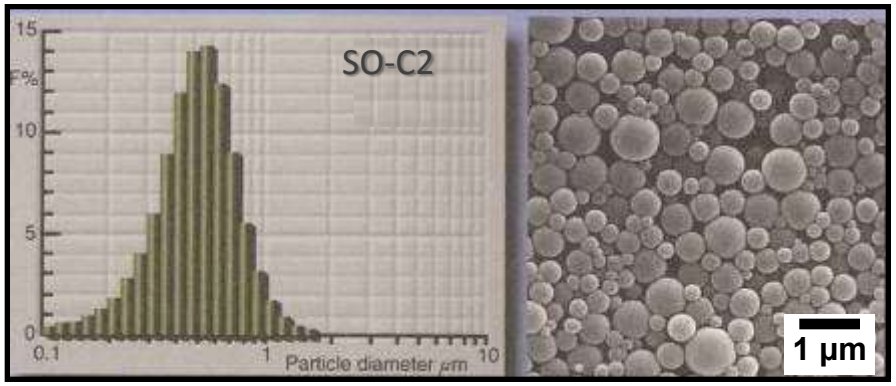
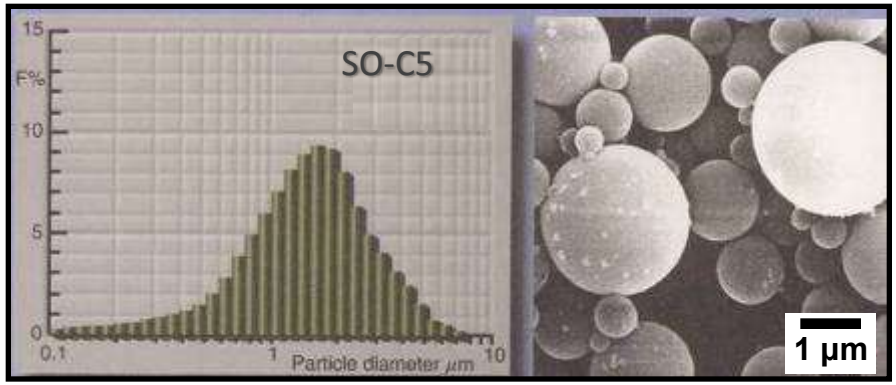
Admafine Silica Line Up for Compounding

	Base Silica Grade							Surface Treatment Grade	
Purity	Basic Grade	Particle Size (µm)	SSA (m2/g)	Cut Level				Treatment	Code
				75µm	45µm	25µm	10µm		
>99.0%	SC2000-SQV	0.5	6.0	SC2000-SQV	-	-	-	Regular Epoxy Vinyl Phenyl Phenyl Amino Metacryl Dimethyl , etc.	-SQ -SE -SV -SP -SX -SM -GD
	SC5000-SQV	1.2	4.5	SC5000-SQV	-	-	-		
>99.8%	SO-C1	0.3	10.0	SC1000-	SC1500-	SC120G-	SC110G-		
	SO-C2	0.5	6.0	SC2000-	SC2500-	SC220G-	SC210G-		
	SO-C4	1.1	4.5	SC4000-	SC4500-	-	-		
	SO-C5	1.5	4.0	SC5000-	SC5500-	SC520G-	-		

※NOTE: This table shows "available products" including grades which are under development.





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Admafine Silica Line Up for Electronics

	Base Silica Grade							Surface Treatment Grade	
Purity	Basic Grade	Particle Size (µm)	SSA (m2/g)	Cut Level				Treatment	Code
				45µm	25µm	5µm	3µm		
Regular type (Purity >99.8%)	SO-C1	0.3	10.0	SC1500	SC1200	SC1050	-	Regular Epoxy Vinyl Phenyl Phenyl Amino Metacryl Dimethyl , etc.	-SQ -SE -SV -SP -SX -SM -GD
	SO-C2	0.5	6.0	SC2500	SC2200	SC2050	-		
	SO-C4	1.1	4.5	SC4500	-	SC4050	-		
	SO-C5	1.5	4.0	SC5500	SC5200	-	-		
High purity type (Purity >99.9%, Low α)	SO-E1	0.3	15.0	SE1500	-	SE1050	SE1030		
	SO-E2	0.5	6.0	SE2500	SE2200	SE2050	SE2030		
	SO-E4	1.0	4.5	SE4500	-	SE4050	-		
	SO-E5	1.5	4.5	SE5500	SE5200	SE5050	-		
	SO-E6	2.0	2.0	-	SE6200	SE6050	-		

※NOTE: This table shows "available products" including grades which are under development.

Admafine Silica Slurry Line Up

	Base Silica Grade				Treatment	Solvent	
Purity	Particle Size (μm)	SSA (m2/g)	Cut Level		Surface Treatment	Solvent	Standard SiO_2 contents
			5μm	3μm			
Regular type (Purity >99.8%)	0.3	10.0	SC1050	SC1030	Regular Epoxy Vinyl Phenyl Phenyl Amino Metacryl , etc.	MEK MIBK Cyclohexanone , etc.	50～70wt%
	0.5	6.0	SC2050	SC2030			
	1.1	4.5	SC4050	-			
High purity type (Purity >99.9%, Low α)	0.3	15.0	SE1050	SE1030			
	0.5	6.0	SE2050	SE2030			
	1.0	4.5	SE4050	-			

※NOTE: This table shows "available products" including grades which are under development.

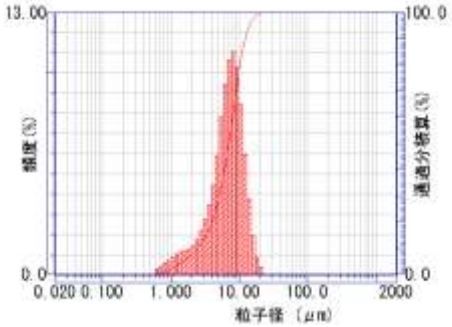
Admafuse Line Up

Purity	Base Silica Grade							Surface Treatment Grade	
	Basic Grade	Particle Size (µm)	SSA (m2/g)	Cut Level				Treatment	Code
				75µm	45µm	25µm	20µm		
High purity type (Purity >99.9%, Low α)	FE9 series	~ 8	2.0 ~	FE975A	-	FE925A	FE920A	Regular Epoxy Vinyl Phenyl Phenyl amino Methacryl Dimethyl , etc.	-SQ -SE -SV -SP -SX -SM -GD
	FEB series	~ 15	1.0 ~	FEB75A	-	FEB25A	-		
	FED series	~ 25	1.0 ~	FED75A	FED45A	-	-		
	FEF series	~ 35	1.0 ~	FEF75A	-	-	-		

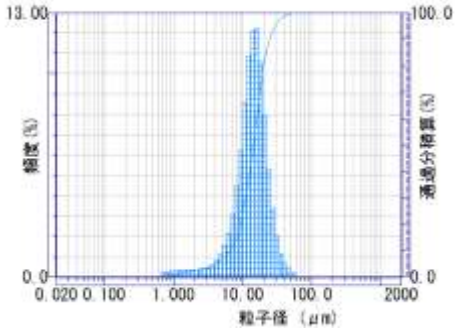
※NOTE: This table shows "available products" including grades which are under development.

※We can provide a product designed for close packing. (PSD and SSA are customizable.)

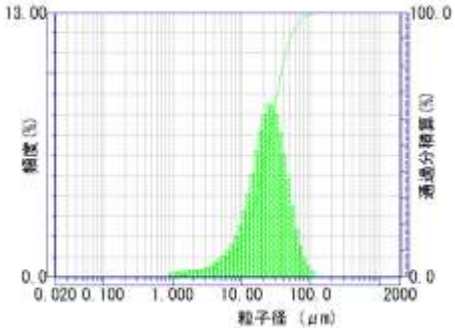
FE9-



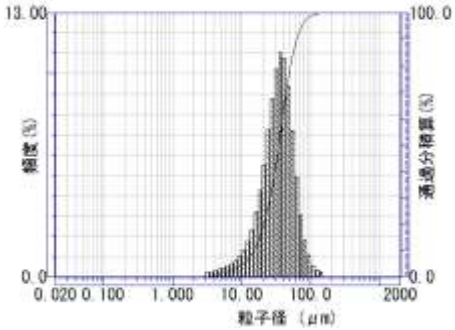
FEB-



FED-



FEF-



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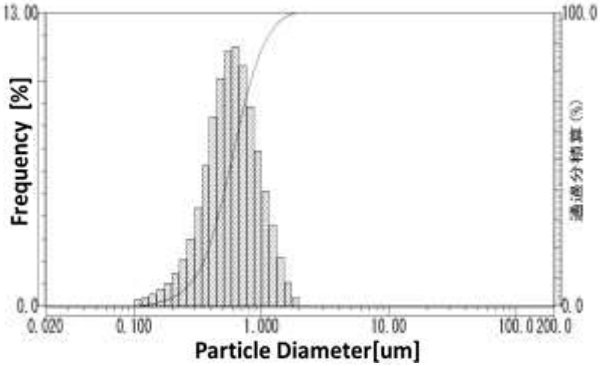
Admafine Alumina Line Up

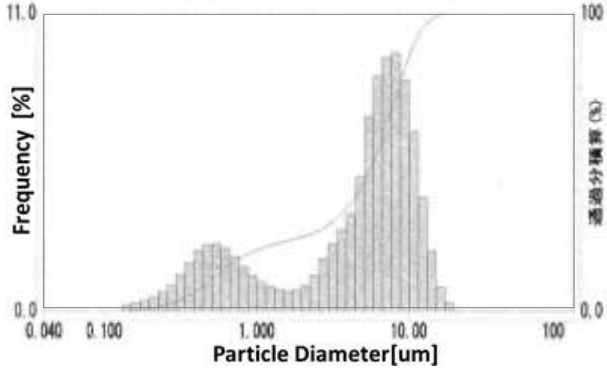
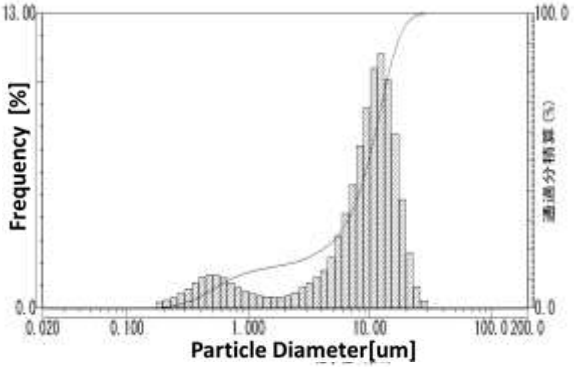
	Base Alumina Grade									Surface Treatment Grade	
Purity	Basic Grade	Particle Size (μm)	SSA (m2/g)	Cut Level						Treatment	Code
				75μm	45μm	25μm	15μm	10μm	5μm		
Regular type (Purity >99.8%)	AO-502	0.6	7.5	AC2000	AC2500	AC2200	-	-	AC2050	Regular Phenyl Phenyl Amino Metacryl Vinyl Dimethyl Silicone , etc.	-SI -SP -SX -SM -SV -GD -SR
	AO-506	6	2	-	-	AC9200	AC9150	AC9100	-		
	AO-509	10	2	AC9000	AC9500	-	-	-	-		
Low α ray type (Purity >99.9%)	AG2050	0.6	7.0	-	-	-	-	-	AG2050		
	AG9200	6	3.5	-	-	AG9200	-	AG9100	-		

※NOTE: This table shows "available products" including grades which are under development.

AO-502 (0.6 μm)



AO-506 (6 μm)



AO-509 (10 μm)