Technology Innovation in the Nanoparticle Project —Synthesis of Nanoparticles and Nanocomposites—[†]

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Abstract

The five-year METI/NEDO's nanoparticle project started in 2001. In this study, various nano-sized particles, e.g. Au, Ag, GaN, ZnO, FePt, CdSe, Y₂O₃:Eu, (Y,Gd)₃Al₅O₁₂:Ce, ZnS:Mn, etc., were prepared by gas-phase methods (thermal and plasma CVD) and by liquid-phase methods (spray pyrolysis, spray drying as well as sol-gel method) using continuous reactors. Nanoparticles and nanoparticle/polymer composite materials were also prepared using polymeric precursor/processing techniques. Using these preparation methods, non-agglomerated and highly-functional nanoparticles were successfully produced in controlled sizes ranging from around 100 nm to a single nanometer with good stoichiometry and high crystallinity.

Keywords: Nanoparticles, Nanocomposites, CVD, Spray Pyrolysis, Sol-gel

1. Introduction

Material synthesis via aerosol and colloidal processing offers a route for production of nanoparticles (with diameters less than about 100nm) of high purity with specifically tailored chemical and physical properties. In particular, single nanometer-sized particles are expected to play an important role in the synthesis of nanostructured materials, as well as in nanotechnology in general, in the 21st century due to unique characteristics such as quantum effects. From this background, the Nanotechnology Particle Project was launched with support provided by the Ministry of Economy, International Trade and Industry (METI) of Japan.

The objective of this project is to establish a platform for developing synthesis and functionalization technologies for nanoparticles, which is important for producing nanostructures and developing nano-functions. The following research themes have been developed for this project: 1) research and development of high-rate synthesis technology for nanopar-

ticles; 2) research and development of dispersion and surface modification technologies for nanoparticles; and 3) preparation and performance evaluation of nanocomposite materials. In this paper, a brief discussion of the research items in the project and the results of the preparation of nanoparticles using aerosol and colloid methods are reviewed.

2. Nanoparticle Synthesis Technologies

In the synthesis of nanoparticles, both gas-phase synthesis and liquid phase synthesis has been used to elucidate the particular features of each method. This was done by investigating the average particle diameter, particle size distribution, morphology/shape, surface characteristics, compatibility with materials and their link with the surface modification techniques. Novel methods of synthesizing nanoparticles were developed by clarifying field conditions such as pressure and temperature and the composition distribution in the nucleation and growth processes of nanoparticles as shown in **Fig. 1**. **Fig. 2** shows the TEM pictures of representative nanoparticles prepared with various methods developed in this project.

2.1 Nanoparticle preparation via gas-phase reaction methods

Material synthesis of nanoparticles via high-temperature processing techniques is a promising tool that offers a good route to production of high-purity

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Fig. 1 Various nanoparticle synthesis methods developed in the project.

Fig. 2 TEM pictures of representative nanoparticles prepared using various preparation methods developed in the project.

nanoparticles with specifically tailored chemical and physical properties. However, the transformation of the gaseous precursor to the final particulate is a complex physical process involving nucleation of the particulate phase, condensation of gasses onto particles, and coagulation and coalescence between particles.¹⁾ Nanoparticle synthesis techniques by CVD methods are roughly classified by heat and energy sources, the state of starting materials, and other factors. However, nanoparticles generated by gas-phase processes are usually in the form of aggregates, due to their coagulation at high temperatures. There have been several recent attempts to combine CVD methods with the ionization of gases or the unipolar charging of particles to generate non-agglomerated nanoparticles. The purpose was to suppress collisional growth of agglomerates by mutual electrostatic repulsions of unipolarly charged particles. Using a newly developed electrospray-assisted chemical vapor deposition (ES-CVD) process, non-agglomerated, spherical nanoparticles of silicon, titanium and zirconium oxides were prepared.²⁾ Non- or only soft-agglomerated nanoparticles are also produced by radio

frequency- and microwave-plasma CVD processes. SiO₂, GaN³⁾ and FePt nanoparticles were produced in this project.

2.2 Nanoparticle synthesis via liquid-phase reaction methods

Many techniques for the preparation of nanoparticles have been developed via the liquid phase route. It is important to develop a synthesis method in which particles have controlled characteristics including size, size distribution, morphology, agglomeration and composition. To be industrially relevant, the process needs to be low-cost and involve continuous operation and a high production rate (10 g/h per reactor, in this project).

Nanoparticles prepared via liquid routes and nanoparticle-based nanostructured materials were investigated. Silica (SiO₂) nanoparticles were fabricated using polymer as a model material for the filler in nanocomposites. The other model materials were phosphors (e.g., Y₂O₃:Eu), luminescent semiconductors (e.g., ZnO, CdSe) and metallic materials (e.g., Au, FePt). Some novel techniques for preparing nanoparticles were developed, especially those focusing on controlling the agglomeration.

FePt nanoparticles have been synthesized from a process involving the mixing of two precursor liquids, ferric acetyl ferric acetyl acetate, Fe(*acac*)₃, and platinum acetyl acetate, Pt(*acac*)₂, in a polyol solution of sodium hydroxide at high temperatures.⁴⁾ The particle size was monodispersed without agglomeration. An ultrasonic field generator also was applied for preparation of FePt and Au. Our group also successfully prepared Au nanoparticles using dendron-grafted phenyleneethynylenes with alpha, omega-disulfur containing groups, which have an intense blue photoluminescence on the composite film.

A continuous-flow reactor has been designed for producing organically capped CdSe nanocrystals as an isolated CdSe nanocrystal, using trioctylphosphine oxide (TOPO) as the capping organic reagent and the high-temperature reaction solvent.⁵⁾ A relatively high reaction temperature (e.g. 350 degree-C) was necessary for matured crystal growth. The quality of TOPO (i.e. impurity compositions such as phosphonic acids) also influenced the quality of the resulting CdSe nanocrystal. The continuous flow reactor produced highly-luminescent, monodispersed CdSe nanocrystals, which was confirmed by transmission electron microscope observation. The production rate was stable for at least 1 h to allow more than 10 g production. This technique was used for the preparation of ZnO nanoparticles surrounded by shell-

like silicate materials and for the preparation of FePt nanoparticles in the project's largest continuous flow reactor (with the rate of 30 g/h).

Synthesis of europium ion doped yttrium oxide ($\text{Y}_2\text{O}_3:\text{Eu}^{3+}$) phosphor nanoparticles, using a relatively high molecular weight polyethylene glycol, has been previously reported. $\text{Y}_2\text{O}_3:\text{Eu}^{3+}$ materials could be prepared by simply heating in air when a water-soluble polymer was added into a solution containing metal nitrates.⁶⁾ When the polymer was absent, flake particles above 2 micrometers in size were formed. A dramatic reduction in particle size occurred when the polymer was added to the precursor. Particles of about 50 nm in size and nearly spherical in shape were observed when $\text{EG}/\text{Y}=0.0475$ and 0.095 mol/mol. The polymer was expected to form carbonaceous materials around the produced primary particles to reduce agglomeration of those particles. The carbonaceous materials could be removed by heating at higher temperatures, resulting in softly-agglomerated particles in the size range of 20-100 nm.

Cerium-doped yttrium gadolinium aluminum garnet, $(\text{Y,Gd})_3\text{Al}_5\text{O}_{12}:\text{Ce}$, was systematically prepared by a polymer-complex method using high-molecular weight polyethylene glycol (PEG).⁷⁾ Well-dispersed nano-sized gallium nitride (GaN) and magnesium-doped GaN (GaN:Mg) particles were successfully prepared from nano-sized gallium oxide (Ga_2O_3) particles in an ammonia gas atmosphere.⁸⁾

In addition to the methods listed above, spray pyrolysis (SP) methods are also promising for nanoparticle preparation, in which the precipitation, thermolysis (i.e. calcination) and sintering stages of powder synthesis can be integrated into a single continuous process. To prepare fine particles by SP, a starting solution is usually prepared by dissolving the metal salts of the product in the solvent. The droplets are atomized from the starting solution with an atomizer, and the droplets are then placed in a furnace. A variety of activities may occur inside the furnace during formation of the final product including evaporation of the solvent, diffusion of solutes, drying, precipitation, reaction between the precursor and surrounding gas, pyrolysis and sintering. Generally, a one-droplet-to-one-product particle (ODOP) conversion is considered the typical particle formation mechanism in conventional spray pyrolysis (CSP). In this project, various modified spray pyrolysis methods were developed and introduced for nanoparticle synthesis, including salt-assisted spray pyrolysis (SASP)⁹⁻¹²⁾ and low pressure spray pyrolysis (LPSP)¹³⁻¹⁸⁾ as well as flame-assisted spray pyrolysis (FASP).¹⁹⁻²⁰⁾

Salt-assisted spray pyrolysis (SASP) is a modified spray pyrolysis method that introduces salts into the precursor solution. It was a versatile method for producing a wide range of nanomaterials, from simple oxide to multicomponent materials. The nanoparticle preparation process by SASP is illustrated in a previous paper by our group.⁹⁾ The investigation of the mechanism of nanoparticle formation by SASP used NiO as a model material. The addition of inorganic salts leads to substantial changes in product particles, and nanoparticles can be obtained under a variety of conditions. Different types of salts and precursors greatly influence the separation process as well as the characteristics of these products, which, along with synthesis temperature, can be used to control the properties of the final product.¹⁰⁾ The capability of SASP in the preparation of simple oxide nanoparticles was also shown in the production of ZnO ¹¹⁾, NiO_2 and CeO_2 . Highly crystalline, dense BaTiO_3 nanoparticles in a size range from 30 to 360 nm with a narrow size distribution also were prepared successfully by the SASP method. KNO_3 salt was employed in the process synthesis.¹²⁾ This shows the capability of SASP in the production of multi-component oxide materials. The crystal phase was transformed from tetragonal to cubic at a particle size of about 50 nm at room temperature. SASP also can be used to produce a high-weight fraction of tetragonal BaTiO_3 nanoparticles down to 64 nm in a single step. The particle size was highly affected by operational conditions. The particle size decreased with decreasing salt concentration, operating temperature and droplet/particle residence time in the hot zones.

Low-pressure spray pyrolysis (LPSP) was applied to the proper nanoparticle synthesis as well.¹³⁻¹⁶⁾ The distinguishing feature of this process is a dramatically different particle formation mechanism. It is expected that a micron-sized droplet will first undergo rapid solvent evaporation upon entering the low-pressure environment. Nucleation and crystallization will be accelerated due to this high evaporation rate. The primary crystals then will undergo Brownian motion inside the droplets. The agglomeration of these primary crystals will be limited due to their very short residence time under the low-pressure conditions. In addition, gas evolution due to thermal reactions and the high drying rate from the furnace will cause some pressure inside droplets/dried particles, which could be the main reason for the dispersion of primary crystals into final nanoparticles. The droplets may break up depending on the properties of the precursor itself. Furthermore, because of the rapid

drying rate at high temperatures, the final particles could be fragmented into multiple nanoparticles, which actually are single primary crystals. Low pressure is considered a driving force for the formation of nanoparticles. Submicron and even micronsized particles may be formed due to the slow drying rate and the physical properties of the precursor. This indicates that the mechanism of particle formation in the LPSP process is obviously complex, including not only process parameters, but also the physicochemical properties of the precursor. Various kinds of materials, from single oxide to multicomponent materials, i.e., nickel,¹³⁾ nickel oxide,¹³⁾ titanium oxide,¹⁴⁾ barium titanate,¹⁵⁻¹⁶⁾ indium tin oxide,¹⁷⁾ and a doped phosphor material ($\text{Y}_2\text{O}_3\text{:Eu}^{3+}$), have been prepared successfully via the LPSP method.¹⁸⁾

Flame-assisted spray pyrolysis (FASP) is another aerosol route for nanoparticle preparation. In this method, a flame aerosol reactor/burner (instead of a traditional electric furnace) allows the input fuel, which provides a high-energy source, to be easily changed and controlled. Aqueous solutions of precursors have been used recently in the flame-spray process for preparation of single and multicomponent nanoparticles by our group.¹⁹⁻²⁰⁾ The use of aqueous precursors is also driven by the low cost of metal salts such as nitrates and acetates and the availability and high solubility of metal salts in water. Submicron and nanosized particles can be produced depending on the operation conditions used and the physicochemical properties of the precursors.

3. Nanoparticle Dispersion & Surface Modification Techniques

For many nanoparticle applications, it is necessary to form a stable colloidal nanoparticle suspension. However, the surface energy of nanoparticles is significantly higher than that of larger particles, so nanoparticles tend to agglomerate in liquid suspensions. Stable nanoparticle suspensions are often formed by adjusting the suspension ionic strength and pH or by surface modification of the nanoparticles themselves. (Fig. 3) Mechanical milling processes, such as bead milling, are an alternative to chemical and surface modifications for making stable nanoparticle dispersions.

A new type of bead mill for dispersing nanoparticles into liquids has been developed.²¹⁾ The bead mill utilizes centrifugation to separate beads from nanoparticle suspensions and allows for the use of small-sized beads (i.e. 15–30 μm in diameter). The

Fig. 3 Dispersion and surface modification techniques in the project.

performance of the bead mill in dispersing a suspension of titanium dioxide nanoparticles with 15 nm primary particles was evaluated through experiments. Dynamic light scattering was used to measure titania particle size distributions over time during the milling process. Bead sizes in the 15–100 μm range were used. It was found that larger beads (50–100 μm) were not capable of fully dispersing nanoparticles, and particles reagglomerated after long milling times. Smaller beads (15–30 μm) were capable of dispersing nanoparticles, and a sharp peak around 15 nm in the titania-size distribution was visible when smaller beads were used. Because nanoparticle collisions with smaller beads have lower impact energy, it was found by X-ray diffraction and transmission electron microscopy that changes in nanoparticle crystallinity and morphology are minimized when smaller beads are used. Furthermore, inductively-coupled plasma spectroscopy was used to determine the level of bead contamination in the nanoparticle suspension during milling. It was found that smaller beads are less likely to fragment and contaminate nanoparticle suspensions. The new type of bead mill is capable of effectively dispersing nanoparticle suspensions and will be extremely useful in future nanoparticle research.

4. Preparation of Nanocomposite Materials

Nanoparticles and nanocomposites are expected to be the next generation of materials, because of a variety of characteristics including their mechanical properties and their optical and heat responses. However, nanoparticles are difficult to handle, especially during industrial production. To control this problem, our group investigated building submicron powders from nanoparticles (with sizes from 4 to 100 nm) us-

ing the spray-drying method.²²⁾ The prepared silica powders have a spherical morphology, with the final size (between 0.1 and 2 μm) controllable by the concentration of the starting colloidal suspension.

Using a similar method, our group successfully prepared microencapsulated powders derived from a precursor of a mixture of two types of sols as well as a mixture of a sol-aqueous solution. Microencapsulated Small-SiO₂/Large-SiO₂, Al₂O₃/SiO₂ as well as ZrO₂/SiO₂ powders were prepared.²³⁾ The light-scattering characteristics of the prepared particles were investigated, and it was found that the refractive index of prepared powders was controllable by their mixture ratio.

Using the spray method, our group investigated the preparation of nanocomposite materials. A mixture of 3 nm of zinc oxide (ZnO) nanoparticles and silica nanoparticle colloid (or TEOS solution) was spray-dried to form a ZnO/SiO₂ powder nanocomposite.²⁴⁻²⁵⁾ The green photoluminescence (PL) exhibited by the composite was very stable. Since the excitation and emission luminescence spectral positions of ZnO are dependent on particle size, composites that emit a specific color can be produced. By using ZnO colloids that have been aged for different times, composites containing ZnO particles of different sizes can be produced. Our group has produced composites that emit colors from blue (460 nm) to yellow-green (550 nm).

4.1 Preparation of porous materials

The synthesis of ordered porous materials presents a fascinating and intellectually challenging problem due to its potential for applications in catalysts, chromatography, controlled-release drugs, low dielectric constants, pigments, microelectronics and electro-optics. Our group have developed the methods to produce silica powders as well as silica films that contained ordered pores.²⁶⁻³¹⁾ The spray-drying method was used to prepare ordered porous silica powders. A colloidal suspension of silica nanoparticles and polystyrene latex (PSL) nanoparticles was mixed and sprayed as droplets into a reactor containing two temperature zones, as shown in our group's previous research.²⁶⁻²⁷⁾ The solvent in the droplets was evaporated at the front part of the reactor to produce a powder composite of silica and PSL nanoparticles. The PSL nanoparticles in the powder were evaporated in the downstream portion of the reactor to produce a silica powder of ordered pores. The pores were arranged into hexagonal packing, indicating that a self-organization process occurred spontaneously during solvent evaporation. The entire

process was completed in only a few seconds, which is superior to the current methods requiring several hours to several days to achieve.²⁸⁾ Furthermore, our group used a similar method to successfully prepare other materials such as silicon dioxide, titanium dioxide, aluminum dioxide, zirconium dioxide and yttrium dioxide particles containing macropores with ordered, hexagonal closed-packing structures.²⁹⁾

Silica films containing a three-dimensionally (3D) ordered pores, with sizes in the range of 40–1000 nm, were produced using a dip-coating method.³⁰⁻³¹⁾ A silicon wafer or glass substrate was dipped vertically in the precursors containing PSL and silica nanoparticles. The PSL particles were then completely removed at a temperature of approximately 400°C. This method permitted the pore size to be selected through appropriate adjustment of the size of the PSL particles. The presence of an optical band gap is dependent on the dielectric constant periodicity of the material. The film produced using this procedure had a dielectric constant as low as 1.192, confirming that the proposed method also has the potential for producing ultralow dielectric-constant materials. When the films were irradiated with a white light source, the reflective spectrum was changed by varying the incident angle, indicating its possible use as a monochromator. As an advanced result, our group successfully produced an ordered, macroporous, iron-platinum (FePt) film as shown in Fig. 3.³²⁾ The prepared film had a magnetic property—a coercivity of up to 10 kOe after annealing at a temperature of 600°C (L1₀ phase).

4.2 Nanoparticle/polymer composite materials

In the past decade, significant progress has been achieved in the synthesis of various types of polymer-nanocomposites and in the investigation of their optical, electronic and magnetic properties. Our group developed a novel method of preparing a nanoparticle-based nanocomposite polymer in which nanoparticle fillers were grown in a polymer matrix. As an example, zinc oxide (ZnO) nanoparticle-based nanocomposite polymer electrolytes with a very high luminescence intensity were prepared by this method.³³⁾ It was found that non-agglomerated ZnO nanoparticles were widely and evenly dispersed in the polymer. A large number of ZnO nanoparticles served as luminescent centers for inducing high luminescent intensity. Compared to conventional preparation conditions, the luminescent intensity was enhanced about 22 times for the ZnO nanopowder.

In another experiment, nanostructured ZnO/Eu and ZnO/Y₂O₃:Eu composites were produced by hy-

dolyzing a mixture of zinc acetate, yttrium acetate and europium acetate in an ethanol solution, followed by mixing with lithium hydroxide.³⁴⁾ The powder produced might be used as filler in luminescent polymer electrolyte composites, which can produce two colors: red (Eu emission) when excited using a wavelength of around 254 nm; and, green (emitted by ZnO nanoparticles) when excited using a wavelength of around 365 nm.

Our group also prepared the hybridization of silica nanocomposites in a thermoplastic resin using a melt extrusion method.³⁵⁾ It was found that uniform dispersion of nanoparticles in the resin can be attained by treating nanoparticle surfaces with an organic compound and modifying the resin prior to mixing, as well as the optimization of the screw design of a twin-screw extruder as shown in Fig. 4. Since the domain size of silica nanoparticles in the resin is smaller than the wavelength of visible light, the obtained composites are transparent in the visible region, which is important for industrial applications.

Fig. 4 Nanoparticle dispersion and nanocomposites preparation techniques.

5. Summary

The preparation of nanoparticles by aerosol, colloid and polymer processes is becoming more and more important. Through the nanoparticle project, various nanoparticles with high quality have been prepared. The evolution of functional elements of nanoparticles and assembling nanoparticles plays an important role in the synthesis of nanostructured materials. By introducing particle engineering and material science, novel materials and new phenomena can be discovered, and guidelines for scale-up in nanomaterial processing will be readily established.

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