Characterization and Technology of Nanomaterials

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Abstract. In order to let more people understand nanomaterials, the common analysis methods and characterization technologies of nanomaterials were summarized. Especially the composition, morphology, particle size, structure, surface and interface analysis of nanomaterials were briefly introduced.

1 Introduction

Nanomaterials refer to materials with at least one dimension in the nanometer scale of the three-dimensional space size (1 to 100 nm), or materials composed of nanostructural units with special nature, known as "one of the most important strategic high technology materials in the 21st century ". Due to the particularity of structure and is thermodynamically unstable state, nanomaterials with special performance of small size effect, surface effect, quantum size effect and macroscopic quantum tunnel effect, and traditional materials do not have many physical and chemical properties, such as high chemical activity and strong adsorption, special catalytic, special optical properties, special electromagnetic properties and hydrogen storage properties. So it is widely used in medical, manufacturing industry, materials, communication, biology, environment, energy, food and other fields. With the expansion and enhancement of the application field, the toxicity and safety of nanomaterials have been widely concerned in recent years [1, 2].

Analytical science is one of the most important and active areas of human knowledge. It is not only the object of study, but also the important means to observe and explore the world, especially the microscopic world. With the development of the nano materials science and technology, the improvement and development of new methods of analysis, new analysis techniques and new concept, improve the sensitivity, accuracy and reliability, to extract more information from, to improve testing quality and efficiency and economy is demanded. Nano science and technology are the properties and interactions of materials (including atoms and molecules) at the nanometer scale (0.1 ~ 100nm). Nano technology is the foundation of the future high technology, and suitable for the research of nanometer science and technology is an indispensable means of the instrument analysis. Therefore, the analysis and characterization of nanomaterials have important significance and effect on the development of nano materials and nanotechnology [1-3].

2 Classification of nanomaterials

There are many kinds of nanomaterials, and the scientific classification is the prerequisite for the characterization of nanomaterials. Table 1 list classification of nano materials based on the external dimensions in China and ISO standards [4].

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Table 1 the clasification of nanomaterials

Space dimension	China	ISO	Examples
Three external dimensions of the material are in nanometer scale	Zero dimensional nanomaterials (0D)	Three dimensional nanomaterials (3D)	Nano particles, nano capsules, quantum dots, fullerene
The two external dimensions of the material are in nanometer scale, and the third external one is larger than 100 nm	One dimensional nanomaterials (1D)	Two dimensional nanomaterials (2D)	Nanofibers, nanotubes, nanowires
One external dimension of the material is in the nanometer scale, and the other two external dimensions are larger than 100 nm	Two dimensional nanomaterials (2D)	One dimensional nanomaterials (1D)	Nano Film, Nano board

As it can be seen in the research field of nanomaterial, usually divided into zero dimensional nanomaterials, one-dimensional nanomaterials and two-dimensional nanomaterials; and in the international standard, but the corresponding three-dimensional nanomaterials and two dimensional nanomaterial and the one-dimensional nanomaterials. This is because our standard called "zero dimensional" and "one-dimensional" and "two-dimensional" refers to the external dimensions of the material not dimensions in the nanometer scale three-dimensional space, and ISO standards referred to in "3D", "two" and "one-dimensional" refers to the external material size dimensions in the nanometer scale three-dimensional space. By comparison, ISO standard nanomaterials classification is more intuitive, easy to understand, it is recommended for the classification of nanomaterials and international unity, the use of ISO standard classification, more conducive to the exchange of scientific and technological information. This paper discusses the basis of classification using ISO Standard Classification

3 Properties of nanomaterials

3.1 Small size effect

When the nanoparticle size and light wavelength, de Broglie wavelength of the conduction electrons and superconducting state coherence length or depth of penetration physical features such as size is, the crystal periodic boundary conditions will be destroyed, sound, light, mechanical, electrical, thermal, magnetic, internal pressure, chemical activity, etc and ordinary particles compared have changed greatly, this is the size effect of nanoparticles (also known as volume effect structure consistent, but because each grain contains only a finite lattice, lattice will happen a certain degree of elastic deformation.

3.2 Surface and interface effects

Nanoparticles because of its small size and surface area, surface energy high, is located in the surface atoms are in serious absence of state, so the activity is very high, is not stable, encounter other atoms very fast binding, this activity is surface effect.

3.3 Quantum size effect

When the crystallite size and the de Broglie wavelength at that time, particles in the electron motion in three directions were restricted, electronic continuous energy band is split to close to the molecular orbital, nanoparticles of sound, light, electricity, magnetic, thermal and superconductivity and macroscopic properties have very different, known as quantum size effect.

3.4 Macroscopic quantum tunneling effect

Tunnel effect refers to the microscopic particles with barrier penetration ability, and later it was discovered that macroscopic quantities, such as magnetization, quantum interference device of the magnetic flux also has a tunnel effect, known as macroscopic quantum tunnel effect. The nanoparticles have a tunneling effect, which is similar to that of microscopic particles. The effect is also called the macroscopic quantum tunneling effect [4, 5].

4 Five kinds of typical nanomaterials

4.1 Nano particle material

Nano particle materials, also known as nano powder, generally refers to the particle size of 100 nm below the powder or particle. Due to the small size, large specific surface and quantum size effect, it is different from the conventional solid. For high density magnetic recording materials, ceiling wave stealth material, magnetic fluid material, anti-radiation material, monocrystalline silicon and precision optical device polishing materials, microchip heat conducting substrate and wiring materials, microelectronics packaging materials, optoelectronic materials, battery electrode materials, solar cell materials, catalysts with high efficiency and high efficient combustion supporting agent, sensitive components, high toughness ceramic materials, human repair material and anticancer agents and so on[6, 7].

4.2 Nano solid materials

Nano solid materials are generally referred to as ultrafine particles with a size of less than 15 nm under high pressure, or a dense solid material formed after a certain heat treatment process.

4.3 Nano membrane materials

Nano membrane is a thin film composed of grain (or particles) in nanometer size, and the thickness of each layer is in the nanometer order of magnitude of the single layer or multilayer film.

4.4 Nano magnetic liquid materials

Magnetic liquid is an organic surfactant, which is coated with a layer of long bonds, which is highly dispersed in a certain base fluid, and the magnetic liquid is stable and has a magnetic liquid. It can be in the external magnetic field as a whole movement, so it has no other liquid magnetic characteristics.

4.5 Carbon nanotubes

Carbon nanotubes are black powder, which is composed of a graphite like carbon atom hexagonal grid. It is generally a multilayer, with a diameter of a few nanometers to tens of nanometers, and a length of several microns or even a few millimeters. The carbon nanotube itself has a perfect structure, which means it has a good performance. Its strength in one dimension can exceed the strength of steel wire, and it has the properties that other materials do not possess: very good electrical conductivity, thermal conductivity and electrical properties. The unique properties of carbon nanotubes have determined the new material.

5 Preparation methods for nanomaterials

The concept of "nano materials" at the beginning of the eighties of the 20th century was officially formed, it has become of material science and condensed state physics research hot spot and the preparation of Science in the research of nano material occupies a very key position. The preparation methods of nano materials are divided into two categories: physical and chemical methods [8, 9].

5.1 Physical methods

5.1.1 Vacuum condensation

Using high frequency induction heating, vacuum evaporation, so the gasification or formation of particles, and then quenched. Its characteristics of high purity, good crystal structure, grain size controllable, but the technical equipment requirements.

5.1.2 Physical crushing

Nano particles were obtained by mechanical milling and electric spark explosion. Its operation is simple, the cost is low, but the product purity is low, the particle distribution is not even [10, 11].

5.1.3 Mechanical ball milling method

5.2 Chemical methods

5.2.1 Sol-gel

Sol gel method is widely used for the preparation of metal oxide nanoparticles.

Precursor with metal alcohol salt or non-alcohol salt can be. The substance is that the precursor is hydrolyzed into sol under certain conditions, and then the gel is prepared. The sol-gel method can greatly reduce the synthesis temperature. With inorganic salt as raw materials, the price is relatively cheap.

5.2.2 Hydrothermal

Hydrothermal method is a high temperature and high pressure reaction environment in high pressure autoclave, using water as reaction medium, which makes it difficult to dissolve or dissolve insoluble material, and the reaction can also be re-crystallized. Hydrothermal technology is carried out in a closed vessel to avoid the volatile components of the component. Under hydrothermal conditions powder by hydrothermal crystallization method, hydrothermal synthesis method, hydrothermal decomposition method, hydrothermal dehydration, hydrothermal oxidation method, hydrothermal reduction. In recent years, the electrochemical method and hydrothermal synthesis method are also developed. The former combines the hydrothermal method with the electric field, and the latter uses the microwave to heat the hydrothermal reaction system. Compared with the general wet chemical method, hydrothermal method can be obtained directly from the dispersion and the crystalline powder with good, does not need high-temperature burning treatment, to avoid the possible formation of hard agglomerates [11, 12].

5.2.3 Spincrossover

Using organic solvent instead of water as medium, similar hydrothermal synthesis principle of nano powder using non water solvent instead of water, not only to expand the scope of application of hydrothermal technique, and can realize the reactions under normal conditions cannot be achieved, including the preparation of metastable structures of the materials.

5.2.4 Micro emulsion

Micro emulsion is usually a transparent, isotropic thermodynamic stabilization system consisting of a surfactant, a surfactant, usually an alcohol, and an oil (usually a hydrocarbon). Micro emulsion, tiny "pool" as the surface active agent and surface active agent composed of single molecular layer formed by micro emulsion particle, its size in a few to tens of nanometers, these tiny "pool" separated from each other, is the micro reactor. It has a very large interface, is conducive to chemical reactions.

5.2.5 High temperature combustion synthesis

Provides the necessary energy induced high exothermic chemical reaction using external, system reacts to form chemical reaction front (combustion wave), chemical reactions in its heat release support rapid, burning wave spread throughout the system. The rapid decomposition of the precursor leads to a large amount of gas release, which avoids the adhesion of the precursor due to melting, and the particle size of the product is reduced. System in the moment to reach several thousand degrees of high temperature, the volatile impurities can be removed [13, 14].

5.2.6 Template synthesis

Synthesis of the voids in the structure of the matrix material as a template. The structure matrix is porous glass, molecular sieve, macroporous ion exchange resin and so on. For example, in the cage of the molecular sieve, the nanometer particles can be obtained with uniform size and periodic structure.

5.2.7 Electrolysis

This method includes two kinds of aqueous solution electrolysis and molten salt electrolysis. This method can be of many usually method cannot make or it is difficult to prepare metal ultrafine powder, especially metal powder with great electronegativity. Oxide ultrafine powder can be prepared. The metal ultrafine powder was prepared by the drum cathode electrolysis method with organic solvent in the electrolyte. The cylinder is placed in the two liquid phase, and is located in the two liquid phase. When the roller is in aqueous solution, the metal is separated from the metal, and the metal is stopped when the organic liquid is rotated, and the metal is coated with organic solution.

6 Characterization of nanomaterials

The characterization of nanomaterials is an objective expression of the properties and characteristics of the nanomaterials, including the size, morphology, structure and composition, and other aspects of characterization, see table 2[7-10].

Table 2 The characterization of nanomaterials

Characteristic	Characterization parameters
Size	Grain diameter, diameter or width, length to diameter ratio, thickness of film
Morphology	Particle morphology, aggregation degree, surface morphology, shape, etc.
Structure	Crystal structure, surface structure, molecular, atomic arrangement of space, defects, dislocation, twin boundary, etc.
Component	Main chemical composition, surface chemical composition, atomic species, valence state, functional groups, etc.
Others	Application characteristics, such as dispersion, rheology, surface charge, etc.

Authoritative standard test is a necessary means to obtain sufficient information, accurate and reliable evaluation of the performance and characteristics of nanomaterials. In order to characterize the nanomaterials scientifically, this chapter summarizes the main testing techniques of nanomaterials at the present stage and its measurement range and method standard.

6.1 Photo correlation spectroscopy, PCS

PCS is based on the Brown motion caused by the particle scattering intensity change frequency of the different size analysis. In liquid, the particle is mainly dominated by Brown, and its velocity depends on the factors such as particle size, temperature and viscosity coefficient. At a constant temperature and viscosity, the size of the nanoparticles can be obtained by measuring the diffusion coefficient of the nanoparticles.

6.2 X-ray diffraction, XRD

X -ray diffraction method can be used for structural analysis, size measurement and phase identification of nanocrystalline materials. The wavelength of nm X between the 0.05~0.25 ray and the crystal in the atomic spacing is quite, when it occurs through the crystal diffraction phenomenon. X-ray ray diffraction broadening method line (diffractometry X, XRD-LB) can be used to measure the grain size of nanomaterials based on the width of the diffraction line and the grain size of the material.

XRD phase analysis is produced on the basis of the nanocrystalline samples of diffraction angle position and X-ray diffraction intensity to determine crystallization of each component, valence state of the crystal phase, crystal structure and various elements in the crystal, bonding state. It can be used for the identification of unknown composition.

6.3 Small angle X-ray scattering, SAXS

Small angle X-ray scattering is an analytical method for the determination of long period structures and the size distribution of nano particles by using X-ray coherent scattering near the point of origin.

SAXS can be used for nanoscale of a variety of metals, inorganic nonmetal, organic polymer powder and biological macro molecules, colloidal solution, magnetic fluids particle size distribution determination; also of nanoscale holes in various materials, segregation and precipitation of equal size were analyzed.

6.4 Electron microscopy

TEM is of the size of the nanomaterial, morphology, surface structure and micro area chemical composition of the most commonly used method, in general, including scanning electron microscopy method (SEM) and transmission electron microscopy. SEM is an analytical technique to observe the surface properties of materials by using the two electron, the back scattered electron and the characteristic X ray generated by the interaction between the incident electron beam and the surface. SEM is magnification can be adjusted continuously, from several times to tens of thousands of times, the samples are simple one by one; but the general requirements analysis is conductive solid samples of non-conducting samples need to be surface evaporation conductive layer.

SEM and energy dispersive spectrometer combined, can meet the needs of the surface micro topography, organizational structure and chemical elements Trinity for isotopic analysis. The energy spectrum instrument can be used for point, line and surface analysis of the surface, the analysis speed is fast, the detection efficiency is high, and the spectral line is good.

6.5 Scanning probe microscopy, SPM

SPM is generated using the measuring probe and the sample surface interaction signal analysis techniques in nanometric or atomic level of material surface atoms and molecules of geometric structure and the physical and chemical properties. Atomic force microscope (AFM) can not only directly observe the morphology and structure of the nanomaterials, but also can control the surface of the material.

6.6 X-ray photo emission spectroscopy, XPS

XPS analysis method on the X-ray as the excitation source, based on nanomaterials surface to be inspired by the electronic has the characteristics of energy distribution (spectrum) and the surface elements are analyzed, also known as chemical analysis photo electron energy spectrum (electron spectroscopy for chemical analysis, ESCA) method.

6.7 Auger electron spectroscopy, AES

AES is by electron gun emitted electron beam ejected from the auger electron on the surface of the material for analysis and development has become important means of elements on the surface of qualitative and semi quantitative analysis, elemental depth distribution analysis and micro area analysis. As the auger electron energy only with atomic orbital energy level, and the energy of the incident electron has nothing to do is and regardless of the excitation source, for specific elements and specific auger transition process, the auger electron energy is specific. Therefore, it is very effective to qualitatively analyze all elements of the surface of the sample except for hydrogen and helium, which is based on the kinetic energy of Auger electron. In addition, AES also has a strong ability to analyze chemical valence state. The analysis range of AES is nm 0.5~2.0, and the absolute sensitivity can reach 10~3 single atomic layer, especially suitable for the surface and interface analysis of nanomaterials.

In addition, there are some other testing techniques and methods for characterization of nanomaterials, such as UV/visible/near infrared absorption spectrum method for gold nanorods characterization (GB/T24369.1), UV visible absorption spectra method for cadmium selenide quantum dot nanocrystal characterization GB/T24370, nanotechnology UV visible near infrared (UV VIS) absorption spectroscopy characterization of single wall carbon nanotubes (ISO/TS with)

7 Nanotechnology and the application of nanomaterials

Due to the properties of small size effect, surface effect, quantum size effect and macroscopic quantum tunneling effect, the nano particles present properties not as the same as conventional materials such as magnetic, light, electricity and sensitivity. Therefore, the nano particles have broad application prospects in the aspects of magnetic materials, electronic materials, optical materials, high density materials, sintering, catalysis, sensing, ceramic toughening and so on[12-15].

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