

A REVIEW ON PREPARATION METHODS OF NANOCOMPOSITES

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ABSTRACT

Nanocomposites are the emerging material in the field of nanotechnology disciplines such as electrical engineering, mechanical engineering, physics, chemistry, biology and material science. Nanocomposite is a multiphase solid material, in which atleast one of the phases shows dimensions in the nanometer range ($1\text{ nm}=10^{-9}\text{ m}$). Nanocomposites are multifunctional materials due to their high transparency, electrical conductivity, increased environmental stability, diffusion constants, mechanical strength, optical quality, heat resistance and recyclability. In this review Various methods of preparation of Nanocomposites will be discussed

I INTRODUCTION

There are various methods followed to produce nanocomposites such as melt processing (1), high speed vibrating milling technique (2), ball-milling method (3), microwave assisted technique (4,5), Solvothermal method (6), hydrothermal method (7), chemical vapour deposition (8), template synthesis (9), sputter deposition (10), co-precipitation technique (11), controlled synthesis (12), wet impregnation method (13), sol-gel method (14), etc.

II Ball Milling Method

Ball milling is an example of top-down method used for the synthesis of nano materials. It is a simple, low cost technique and all types of materials can be produced by this method. Mostly, ceramics and metallic nanomaterial can be prepared by this method. These mills consists of wolfram carbide or steel which acts as grinding media. These ball mills rotates with high energy around a horizontal axis inside a drum and crush the solid material into nanocrystallites. This method is not suitable for non-oxides. Metal oxides like Zinc Oxide (ZnO) and Cerium Oxide (CeO_2) can be prepared by this method. It is an attractive technique of solvent free synthesis. Figure 1.4 shows schematic representation of the principle of ball milling method.

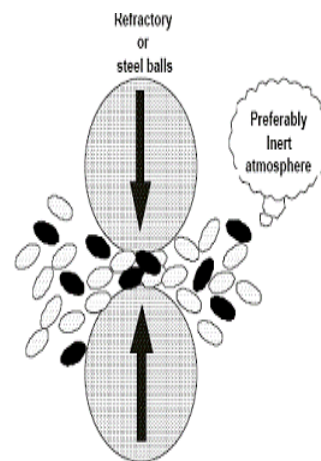


Figure 1. Schematic representation of the principle of mechanical milling

II Solvothermal Synthesis

A solvothermal synthesis is a method of growing single crystals in a closed reaction vessel (Autoclave) at a high temperature greater than the boiling point of the solvent at high pressure (10^5 Pa). Solvothermal means the precursor solvent may be aqueous and non-aqueous. It is similar to the [hydrothermal](#) synthesis route, but the solvent used for the hydrothermal synthesis is water. Using this method, one can gain the benefits of both the hydrothermal and [sol-gel](#) routes. The size, shape distribution and crystallinity of metal oxide nanostructures are greatly controlled by this method. The reaction temperature, time, solvent type, precursor type and surfactant type have played an important role on the properties of the nanomaterials. Solvothermal method is used in laboratory for the synthesis of nano titanium dioxide

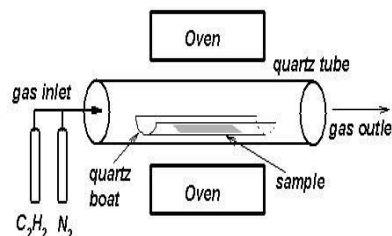
(TiO₂), graphene, carbon and other materials. In recent years, fluorides, nitrides, hybrid materials such as inorganic/biological and inorganic/organic materials with specific properties can be prepared by this method.

III Chemical Vapor Deposition (CVD)

Chemical vapor deposition (CVD) process involves the deposition of solid thin-film to the target surfaces. It is also used to synthesis high purity bulk powders, bulk materials as well as fabricating nanocomposite materials via infiltration techniques. The precursors may be solid, liquid or a gaseous form at ambient conditions but it is converted into vapor before it transferred into the reactor. These vapour precursor is delivered to the hot wall reactor under ambient temperature and it is allowed to deposit on the hot substrate. They react or decompose into solid phase which deposits on the substrate. CVD is a preferred method to process any metallic or ceramic compounds such as borides, nitrides, metals, alloys, Oxides and intermetallic compounds.

Since 1960, CVD has been used to produce carbon filaments and fibers. In this method, a carbon-containing gas is decomposed to a high temperature (600°C or higher) and deposits on a substrate in the presence of catalyst metal particles as shown in Figure 1.5. The lower temperatures used in this method reduce production costs. Carbon nanotubes (CNTs) fabricated by this method have a large number of defects.

Chemical Vapour Deposition technique is mainly uses in coatings, corrosion resistance, wear resistance and erosion protection. It has an application for the synthesis of catalysts, nanopowders and fibers. It is also used in the fabrication of semiconductor devices such as integrated circuits, optoelectronic devices and sensors.



(Source: Marcio R. Loos 2017).

Fig.2 Schematic diagram of the CVD apparatus

IV Sputter Deposition

It involves the ejection of atoms from a target material onto a substrate and they condensed to form a film. The sputtering atom should possess the higher kinetic energy ($\gg 1\text{eV}$). The sputtering gas is often an inert gas such as Argon (Ar). For efficient momentum transfer, the atomic weight of the sputtering gas should be close to the atomic weight of the target, so far sputtering light elements Neon (Ne) is preferable while for heavy elements Krypton (Kr) or Xenon (Xe) are used. In sputtering process (shown in Figure 1.6), the composition of final product is the same as the composition of initial precursor material. This method is highly suitable for non-agglomerated and ultra pure metals.

One of the most important applications of sputtering is the production of computer [hard disks](#), CDs and DVDs. This method is widely used in [semiconductor](#) industry for thin film deposition of various materials in [integrated circuit](#) (IC) processing. Sputtering process is also used in the manufacturing of thin film transistor and [photovoltaic](#) solar cells.

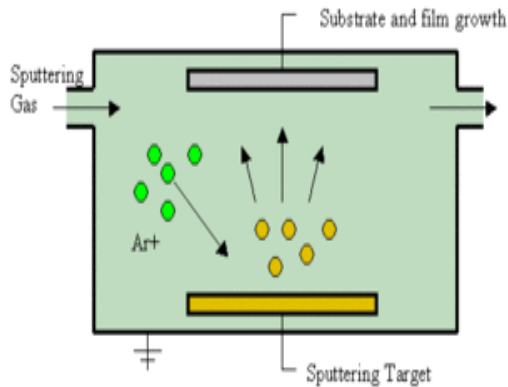


Fig.3 Schematic diagram of Sputter deposition

V Co-precipitation

In a co-precipitation method, the final product is obtained by subsequently calcined the precipitate of a substance at appropriate temperature. The desired properties of final product can be obtained by controlling the temperature, concentration of solution, PH and stirring speed of mixture. In this method, the reactant mixes homogeneously, a precipitate is formed which reduces the reaction temperature. By calcining the precipitate at suitable temperature, a powder product is obtained. A fine metal nanopowder product at low temperature can be directly synthesized by this method. Co-precipitation is also used as a method of magnetic nanoparticle synthesis.

VI Sol-gel Method

Sol-gel method is a novel chemical route to produce ceramics and ceramic nanocomposites. It is a simple and low temperature synthesis technique with a wide range of structural and micro-structural applications. The materials derived from sol-gel technique have formed wide range of applications in microelectronics, solar-cells, intelligent coatings and batteries as well as in medical field. In the beginning, researchers have focused the sol-gel method to produce silica and silicate glasses but they have progressively established this technique for the production of many oxides, non-oxide ceramics and composites. Using this method one can produce materials in the forms of bulk, fibers, sheets, coating films and particles at relatively low temperature. New

composites with high purity and high homogeneity can be produced by this method. An outline of the sol-gel method is shown in Figure 1.7.

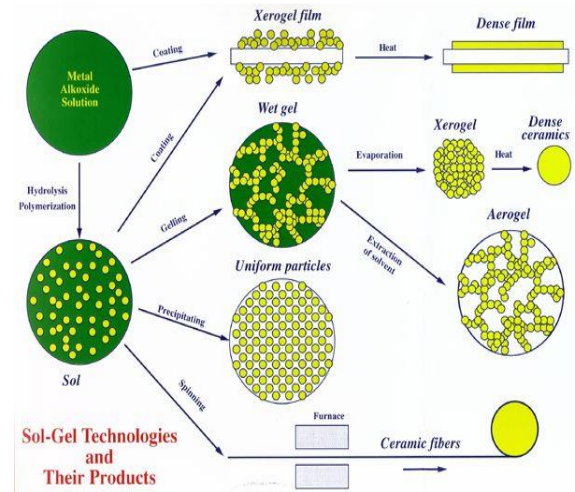


Fig.4 Sol-gel Process

Sol-gel is a useful self-assembly process especially for the fabrication of metal oxides. Sol - a colloid that is suspended in a liquid, gel - a suspension that keeps its shape. Thus sol-gels are suspensions of colloids in liquids that keep their shape

Sol-gel formation occurs in four steps,

1. Hydrolysis
2. Condensation and polymerization
3. Growth of particles
4. Agglomeration

Usually metal alkoxide ($M(OR')_n$) is used as a precursor material in sol-gel process. 'M' is the metal, 'O' is the oxygen and 'R' is the organic group. By adding a small amount of water, hydrolysis takes place. Since water and metal alkoxide are insoluble in each other, they are dissolved in a common alcoholic solvent in order to carry out the reaction. In the hydrolysis reaction, the hydroxyl group (OH)

replaced the alkoxide groups (OR'). Polycondensation reaction produces a gel, a three dimensional network. The structure of gel is purely dependent on water and the catalysis used.

The wet gel, obtained by the hydrolysis and polycondensation reactions, itself is not an end product. In fact, it is necessary drying stage and a suitable thermal treatment in order to obtain the material, glass or ceramic, with the required characteristics.

In the drying process, the wet gel is heat treated for a particular time that allows desorption of water and residual alcohol physically linked to the polymeric network.

Many difficulties arise during the drying stage, mainly caused by the removal of large amounts of solvent trapped in the polymeric network. The transformation of the wet gel to dried gel leads to a volume decrease and it is often associated with the formation of cracks. To minimize these effects, gels are dried by slow heating rate and for coatings; the thickness usually must not exceed 10 μm .

The dried gel is annealed to remove the residual organic groups. During these heat-treatments, condensation reactions among residual alkoxides groups, both on surface and inside the gel, can take place. Successively, suitable heat-treatments allowed obtaining the desired final material.

CONCLUSION ;

Every method has its own pros and cons. Moreover some of the methods are application specific and some are difficult and cumbersome . Out of all the methods discussed here Sol-gel method possesses many advantages than the low temperature process. Materials of larger surface area can be produced by this method by controlling the porosity. It allows the dopant to disperse uniformly into the final product. It is an easy, cheap and convenient method and

considered to be more suitable for the preparation of multicomponent materials

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