

A Review On Wet Chemical Synthesis of Nanomaterials: Sol-Gel Process

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ABSTRACT-Nanoscience primarily deals with synthesis, characterization, exploration, and exploitation of nanostructured materials. There are various methods involved in synthesis of nanomaterial out of which wet chemical method i.e Sol-Gel process is discussed in this paper. Sol-gel is a chemical solution process used to make ceramic and glass materials in the form of thin films, fibers and powders. This process involves the formation of inorganic networks through the formation of a sol and gelation of the sol to form gel.

Keywords: nanomaterials, synthesis, nanotechnology, Sol-Gel process

I. Introduction:

Nanomaterials are part of nanoscience and nanotechnology. A nanometer is one millionth of a millimeter - approximately 100,000 times smaller than the diameter of a human hairs as shown in Fig.1. Nanomaterials are of interest because at this scale various unique and important properties emerge. These emergent properties have the potential for great impacts in various engineering fields.



Fig.1 Nanomaterial(Carbon Nanotube)

2. Methods for creating Nanostructure:

Following are various methods used for creating nanostructure:

- (i) Mechanical Grinding
- (ii) Wet Chemical Synthesis : Sol-Gel Process
- (iii) Gas Phase Synthesis
- (iv) Plasma Processing & many others.

3. Wet Chemical Synthesis Method: Sol-Gel Process:

Basically the wet chemical synthesis of nanomaterials can be classified into two following groups:

1. The top down method: Where single crystals are etched in an aqueous solution for producing nanomaterials, For example, the synthesis of porous silicon by electrochemical etching.
2. The bottom up method: Consisting of sol-gel method, precipitation etc. where materials containing the desired precursors are mixed in a controlled fashion to form a colloidal solution.

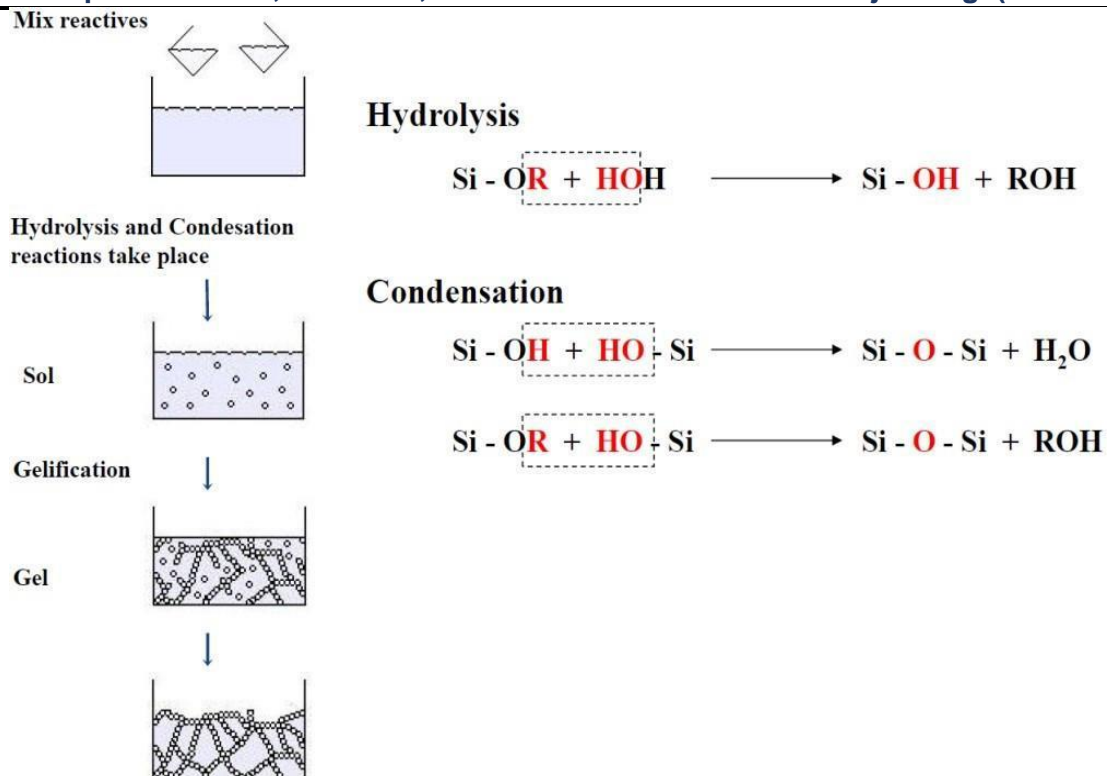


Fig.3 Making Gel Formation

3.1 Sol Gel Process: Techniques and Products

The sol-gel process, involves the evolution of inorganic networks through the formation of a colloidal suspension (sol) and gelation of the sol to form a network in a continuous liquid phase (gel) as shown in Fig.2.

3.1.1 Method of Processing Sol-Gel:

1. The sol-gel process is a wet chemical process that uses either a chemical solution(sol short for solution) or colloidal (sol for nanoscale particle) to produce gel.
2. Metal alkoxides and metal chlorides are typical precursors. They undergo hydrolysis and polycondensation reactions to form a colloid. The sol evolves then towards the formation of an inorganic continuous network containing a liquid phase (gel).
3. Formation of a metal oxide involves connecting the metal centers with oxo(M-O-M) or hydroxo(M-OH-M) bridges, therefore generating metal-oxo or metal-hydroxo polymers in solution.
4. After a drying process, the liquid phase is removed from the gel. Then, a thermal treatment(calcination) may be performed in order to favor further polycondensation and enhance mechanical properties.Refer Fig.4 and Fig.5.

The reactions involved in the sol-gel chemistry based on the hydrolysis and condensation of metal alkoxides $\text{M}(\text{OR})_z$ can be described as follows:



Sol-gel method of synthesizing nanomaterials is widely employed to prepare oxide materials The sol-gel process can be characterized by a series of distinct steps.

1. Formation of different stable solutions.
2. Gelation resulting from the formation of an oxide- or alcohol- bridged network .
3. Aging of the gel (Syneresis), during which the polycondensation reactions continue until the gel transforms into a solid mass, accompanied by contraction of the gel network and expulsion of solvent from gel pores.
4. Drying of the gel, when water and other volatile liquids are removed from the gel network. If isolated by thermal evaporation, the resulting monolith is termed a xerogel. If the solvent (such as water) is extracted under supercritical or near super critical conditions, the product is an aerogel.
5. Dehydration
6. Densification and decomposition of the gels at high temperatures ($T > 8000\text{C}$).

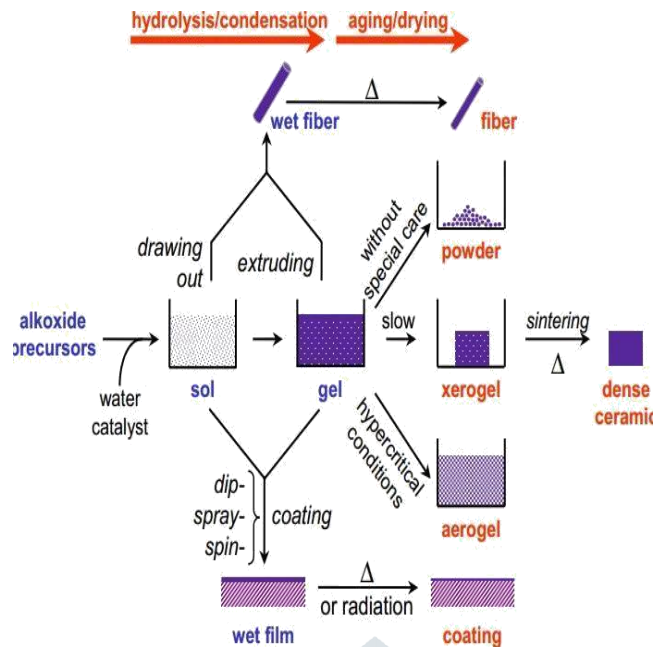


Fig.4 Sol-gel process, techniques and products

The major difficulties to overcome in developing a successful bottom-up approach is controlling the growth of the particles and then stopping the newly formed particles from agglomerating. Other technical issues are ensuring the reactions are complete so that no unwanted reactant is left on the product and completely removing any growth aids that may have been used in the process. The main advantage is one can get monosized nano particles by any bottom up approach.

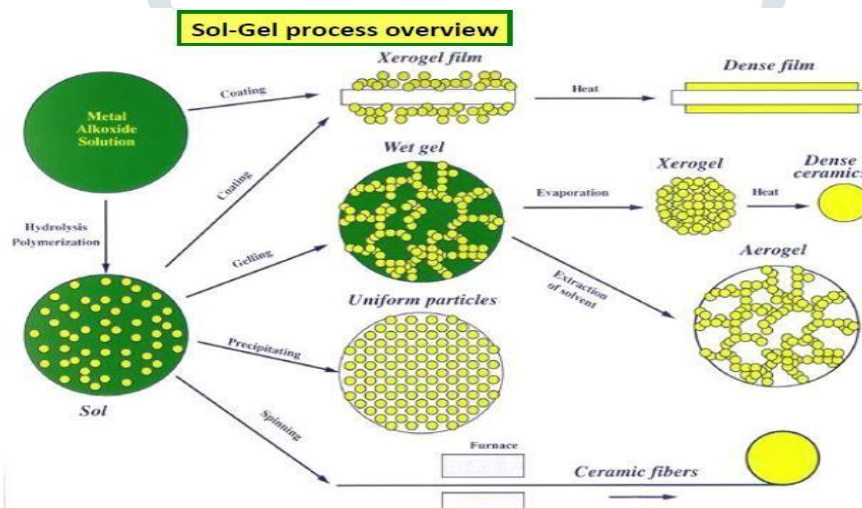


Fig.5 Sol-gel process overview

4. Advantages of Sol-Gel Process:

1. Can easily shape materials into complex geometries in a gel state.
2. Can produce high purity products because the organo-metallic precursor of the desired ceramic oxides can be mixed, dissolved in a specified solvent and hydrolysed into a sol and subsequently a gel, the composition can be highly controllable.
3. It is a cheap and low temperature techniques that allows for the fine control on the product's chemical composition.
4. It has high uniformity.
5. This method is able to get uniform and small sized powder.

5. References

- [1] Zhong Lin Wang, Yi Liu, Ze Zhang (2003), *Handbook of nanophase and nanostructured materials*, Synthesis II, Kluwer Academic/ Plenum Publishers.
- [2] Guozhong Cao [2004], *Nanostructures & Nanomaterials*, Synthesis, properties & applications, Imperial College Press.
- [3] *Nanoscale Science and Technology* (2005) Ed. by R.W. Kelsall, I.W. Hamley, M. Geoghegan. John Wiley & Sons, Ltd.
- [4] *Nanomaterials Handbook* (2006). Ed. by Yu. Gogotsi. Taylor & Francis Group, LLC, 2006. – 2000. – V.49, N6. – P. 705–814.
- [5] G. Scholz E. Kemnitz (2017), Sol-Gel Synthesis of Metal Fluorides, Elsevier-Modern Synthesis Processes and Reactivity of Fluorinated Compounds, pg.no.609-649.