Original Research Article

Synthesis and Application of Carbon Matrix Composites

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ABSTRACT

This paper describes the development of carbon-based composites, and describes the synthesis and application of four carbon-based composites of graphene, carbon nanotubes, fullerenes and graphite.

KEYWORDS: carbon-based composites, graphene, carbon nanotubes, fullerenes, graphite alkynes

1. Introduction

Composite material is composed of two or more different substances through a certain way to combine together to form another multi-phase material. Generally in the composite material is a continuous, called the matrix, the other phase is dispersed, called the reinforcement material. The composite material concentrates on the advantages of the substrate and the reinforcing material showing a unique property.

As the name suggests, carbon-based composite material is carbon fiber (fabric) or silicon carbide and other ceramic fiber (fabric) as a reinforcement, carbon-based composite materials. It is a new type of high temperature resistant material developed in the 1960s, which is composed of reinforced carbon and matrix carbon. The reinforcing carbon may be a different type of carbon (or graphite) fiber and its fabric, acting as a skeleton and a reinforcing agent in the carbon / carbon composite; the matrix carbon may be bonded, and the current matrix carbon may be resin carbon, Carbon and sedimentary carbon. Carbon and carbon composites maintain the advantages of carbon (graphite), overcome the shortcomings of carbon (graphite), greatly improve the toughness and strength, reduce the thermal expansion coefficient, with a small proportion, high chemical stability, good heat resistance (non-oxidation Sexual atmosphere), conductive, good thermal conductivity, good corrosion resistance and so on.

Carbon-based composites have the advantage of being two or more composites, which are unmatched by a single material. Therefore, carbon-based composites have a wider range of applications than single materials, which also give rise to a strong interest in carbon-based composites The In recent years, the discovery of new carbon materials represented by graphene and nitrogen-doped graphene has made carbon materials and carbon-based composites a material star with a more unique nature and a wide range of applications.

1.1. Development

Carbon / carbon composites came out about 1958. Initially, the experimental staff of the Chancewater aircraft company's aerospace branch were accidentally discovered due to an accident when studying phenolic resin-based composites. About the same period, the United States Union Carbide Corporation (UCC) with graphite cloth reinforced resin, after curing, carbonization and graphitization, made the first block of carbon / carbon composite materials and as a commodity for sale.

The development of carbon / carbon composites represented by the United States can be broadly divided into three phases:

The first stage: From 1958 to mid-sixties as the initial stage of development. Mainly in the carbon / carbon composite materials used carbon fiber, carbon / carbon basic composite process research work. To find the carbon fiber elastic modulus and strength of the technical key. In 1965 began to introduce chemical vapor deposition method carbon / carbon of the composite process.

The second stage: The mid-sixties to the early seventies is the carbon / carbon composite materials began to use the stage. During this period, a great deal of work has been done on the enhanced carbon, matrix carbon and composite processes. The carbon / carbon composites have been used as a heat and thermal structural material for cutting-edge technology such as rocket engine nozzles, satellites and spacecraft.

The third stage: Since the early seventies is the development stage of carbon / carbon composites. The properties of the composites were further improved, the anisotropy of the materials was successfully solved, and the three-dimensional orthogonal carbon / carbon composites were developed. The application properties of carbon / carbon composites (anti- Thermal shock) and microstructure were studied. Carbon / carbon composites were studied extensively and deeply as a strategic nuclear weapon head heat-resistant material. MK-12A carbon / carbon nose was successfully used as the third generation intercontinental missile Warheads.

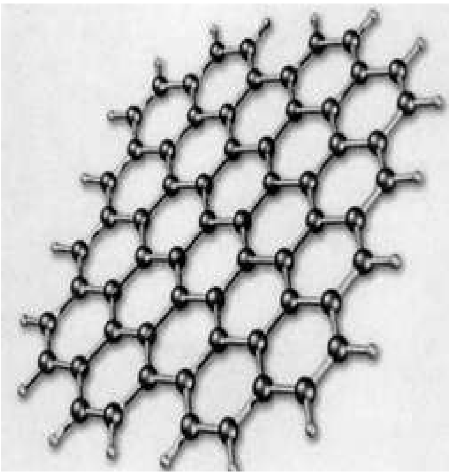
1.2. Preparation

Dipping method: That is, with the impregnated molten oil or coal tar pitc, and then by carbonation and graphite treatment. Its matrix is graphite carbon was layered structure, the performance is anisotropic. There are also impregnated with thermosetting resin such as furfuryl alcohol or phenolic resin which is treated only by carbonization. Its matrix is glassy carbon, that is, amorphous carbon structure and the performance is isotropic.

CVD method: That is, the pyrolytic carbon of the hydrocarbon compound is deposited on the reinforcement to compound, and the carbon matrix of this method is pyrolytic carbon similar to glassy carbon. Carbon / carbon composites are not resistant to oxidation and sometimes need to add antioxidant coating.

2. Graphene

The hybrid form of the carbon atom in the graphene is sp2, and the covalent bond with an angle of 120 degrees is connected with the other carbon atoms into a hexagonal layered honeycomb structure. In addition, each carbon atom has a pz. The number of coordination atoms in the layer is 3, and the bond length between adjacent carbon atoms is 0.142 nm.



Graphene is the base of zero-dimensional fullerenes, one-dimensional carbon nanotubes and three-dimensional graphite. If the five-membered ring in the graphene plane lattice will bend the graphene plane, it will form a richler when there are more than 12 rings. Carbon nanotubes can be seen as crusted by graphene, and graphite can be seen as composed of graphene through the van der Waals stack.

2.1. Synthesis of graphene

Graphene has many excellent physical properties, for how large-scale, high-quality preparation of monolayer graphene is now scientists face an important research content. The main methods for preparing graphene so far are: mechanical stripping, chemical vapor deposition, epitaxial growth, chemical oxidation and reduction and other methods [1].

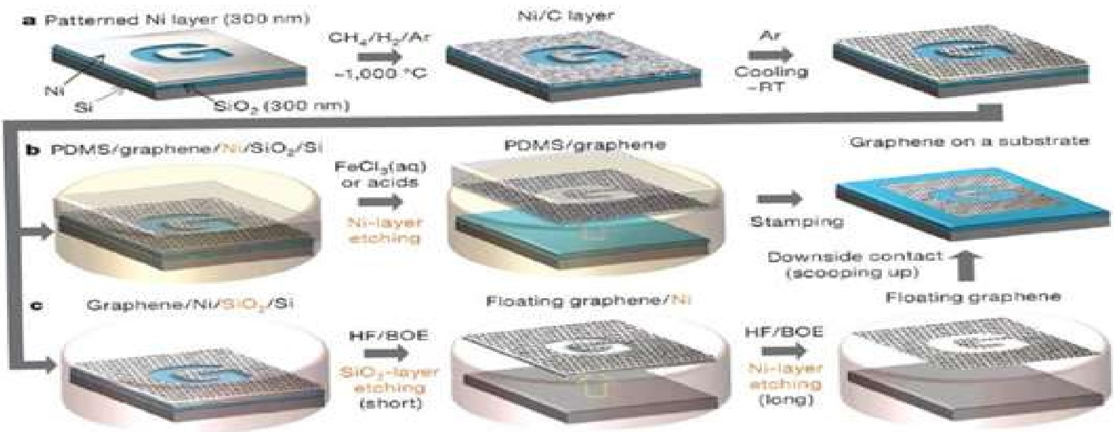
2.1.1 Mechanical stripping method [1-3]

The mechanical stripping method was developed by the Geim Professor Group at the University of Manchester in the United Kingdom in 2004 to develop a graphene method. They use a transparent tape repeatedly paste the graphite, and then with a graphite sheet of adhesive tape affixed to the substrate such as silicon, and then dissolved with acetone tape and then monolayer and less graphene transferred to the substrate such as silicon [2].

In addition to the use of micro-mechanical stripping method for the preparation of graphene, there is a more mature mechanical stripping method - liquid phase ultrasonic stripping method. Liquid phase separation method refers to the bulk of graphite placed in organic solvents such as N-methyl pyrrolidone (NMP) or o-dichlorobenzene for ultrasound, monolithic and multi-layer can be obtained in the coexistence of graphene dispersion [1].

2.1.2 Chemical Vapor Deposition [4]

Chemical vapor deposition (CVD) principle is in the high temperature decomposition of gas (CH4, C2H2), through high temperature annealing makes carbon atoms deposited on the surface of the substrate to form a single layer of graphene. This method is controllable and can produce graphene in large area.



2.1.3 Epitaxial growth method [5,6]

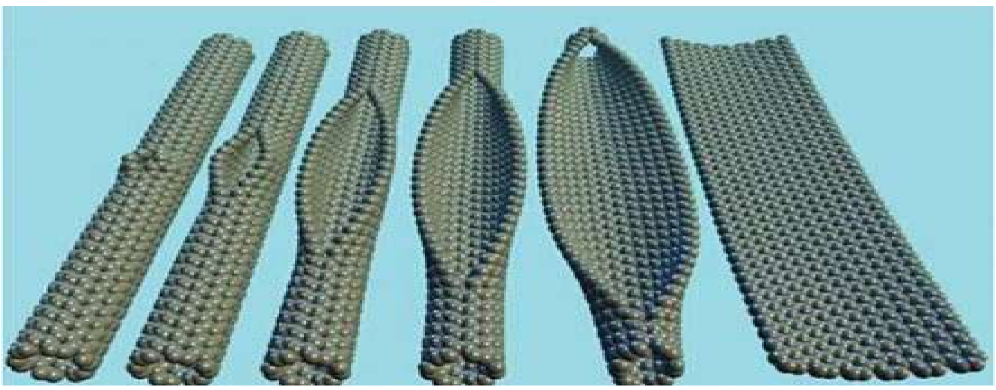
Epitaxial growth means that the silicon is removed by high temperature heating under ultra-vacuum conditions, and the carbon atoms are rearranged to form a graphene sheet.

Heer et al. [7] used silicon carbide as the raw material to evaporate silicon atoms from silicon carbide at 1300 ° C and 1.33 × 10-10 Pa high vacuum to obtain a continuous large area of graphene film. The epitaxial growth method can produce high quality graphene, but there are still some problems in controlling the number of layers and large area of graphene.

2.1.4 Chemical oxidation reduction method [3]

The Reduction of Graphene Oxide Solution is the most widely used method for the preparation of graphene. The method is cheap and the preparation process is simple. Chemical oxidation reduction method for the preparation of graphene has three processes: graphite oxidation → stripping → reduction, as shown below.

2.1.5 Preparation of graphene nanobelts from carbon nanotubes [1]



Due to the limited band gap of two-dimensional graphene, the application in many fields such as field-effect transistors is limited. It is found that quasi-one-dimensional graphene nanobelts (GNRs) have bandgap. The GNRs can be prepared by ultrasonic or chemical shear of two-dimensional graphene sheets, but the nanobelts obtained are not controllable [8]. Dai and so on through the chemical etching technology to cut MWNTs successfully prepared GNRs, first WMNTs deposited on the Si matrix, and then coated with a layer of MWNTs 300nm PMMA film, followed by the use of Ar plasma etching MWNTs by controlling the etching Time to control the number of GNRs, and finally remove the polymer components to obtain GNRs with a width of 10-20 nm. The GNRs obtained have semiconductor properties and have a wide range of applications in the electronics industry. James and so on using potassium permanganate and sulfuric acid as oxidant, in the more moderate conditions along an axis gradually cut MWNTs and also obtained GNRs. The preparation of the schematic and mechanism shown in the following figure. Although GNRs prepared by oxidation are not semiconductors, this method is simpler and more efficient and easier to produce on a large scale.

2.2. Application of graphene

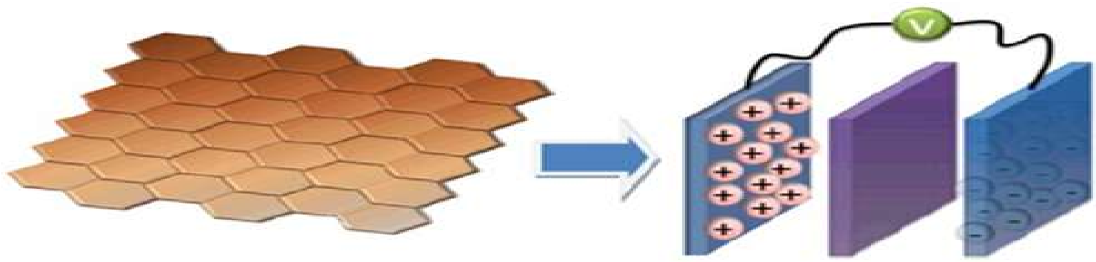
2.2.1 Transparent conductive electrode

At present, the most commonly used material for transparent conductive electrodes is indium doped tin oxide (ITO). ITO is widely used with mobile phone touch panels, monitors and solar cells, but indium is expensive for precious metals and difficult to recover. The graphene has the advantages of light weight, high strength, excellent flexibility and conductivity. At the same time, because of the special structure of graphene has a higher light transmission, quickly become the ideal material instead of ITO [3].

Wang et al. used graphene as a transparent conductive film in a solar cell with a conductivity of 550 S / m and a conversion efficiency of 0.26%. Peking University Liu Zhongfan Study Group made graphene electrode by reduction and repair of graphene oxide, the conductivity reached 350-410S / cm, and the light transmittance was 95%. Geng et al. prepared a transparent graphene conductive film by a two-step reduction method and applied it to an organic solar cell. The efficiency of the whole device was about 1.01%.

2.2.2 Super Capacitors [3,9,10]

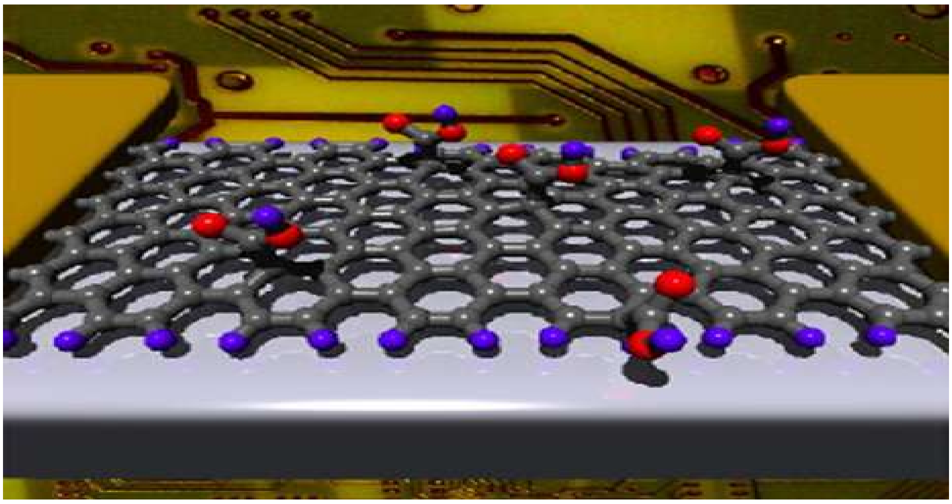
Super capacitor is between the capacitor and the battery between the energy storage device, is the transmission of energy and energy storage system. The specific surface area of graphene is very large and the conductivity is excellent, and graphene is stable in high conductivity while ensuring the sweep voltage. The large specific surface area of graphene can increase the aggregation capacity of electrons and can be used as the capacity of supercapacitors. The use of graphene as an electrode greatly reduces the charging time and increases the capacity by 5-6 times.



Due to the special structure of graphene, the researchers have done an in-depth study of supercapacitors. Stoller et al. Used monolayer graphene as the electrode for supercapacitors, which had a specific capacitance of 135 F / g in water and a specific capacitance of 99 F / g in the organic electrolyte. Le et al. Used a new method of directing the graphene solution onto the titanium mesh, and then preparing a supercapacitor of graphene film by thermal reduction. After 1000 cycles of voltammetry, the specific capacitance decreased from 125F / g to 121F / g, and the loss rate was less than 3%. Ning et al. prepared a controlled structure of graphene web by template chemical deposition method. The thickness of graphene web was 1-2 layers and the specific surface area was 1654m2 / g. The polystyrene net was maintained at 94.1% after the 2000 volt-volt cycle with a specific capacitance of 245 F / g in the KOH solution as a supercapacitor electrode. Yoo et al. prepared a graphene-based planar structure of supercapacitors, the mass ratio of the capacitor can reach 250F / g while the area than the previous reported capacitance reached 394F / cm2.

2.2.3 Sensor [3]

The carbon atoms in the graphene are very flexible to each other and form a large π-bond in the honeycomb. When strongly applied to the surface of the graphene, the surface of the graphene will bend, but the carbon atoms of the graphene surface do not need to be rearranged to accommodate the effects of external forces. And the structure of the graphene makes the graphene excellent in electrical conductivity. As shown below, for the graphene gas sensor.



In 2007, Professor Novoselov et al. successfully prepared a graphene-based sensor with high sensor sensitivity to test the resistivity in a short time. (CO, NH3, NO2) adsorbed on graphene for effective research. Fowler et al. used high-quality graphene by oxidation-reduction method, and successfully prepared a graphene-based sensor. In addition to gas detection, but also on the dinitrotoluene for effective detection. Zhang et al. found that doping graphene, in the preparation of sensors will improve their own sensitivity and select the accuracy. In the process of graphene preparation by chemical vapor deposition on the surface modification of Pd particles, the sensor of the H2 detection sensitivity greatly improved.

3. Carbon nanotubes

The carbon atoms in the carbon nanotubes are mainly sp2, but the cylindrical bending structure leads to quantum confinement and σ-Π re-hybridization, where the three σ bonds are slightly deviated from the plane and the off-bound Π orbit is more biased. The carbon nanotubes are divided into single-walled carbon nanotubes (SWNTs) and multi-walled carbon nanotubes (MWNTs).

3.1. Synthesis of Carbon Nanotubes [11]

At present, the preparation of single-walled carbon nanotubes has only yield levels, and it is still difficult to control single-walled carbon nanotubes with the desired structure. In contrast, the preparation of multi-walled carbon nanotubes is more mature, yield up to kg / h, and can control the diameter and growth orientation of carbon nanotubes. Arc method, laser ablation method, vapor deposition method is the more widely used method.

3.1.1 Arc method:

Laboratory arc synthesis device is generally in the lower than the atmospheric pressure Ar gas water cooled cavity, the horizontal or vertical placed two diameter of 6-12mm, the gap of 1-4mm graphite rod for the electrode, the DC discharge between the electrodes Arc. The carbon nanotubes produced by the arc process are less and less defective than the carbon nanotubes produced by other methods, and therefore have higher mechanical strength and better thermal conductivity.

The preparation of single-walled carbon nanotubes by arc method requires the drilling of an axial hole in the graphite rods filled with dense metal and graphite mixtures as the anode. There are a variety of metal or mixture catalysts to fill graphite rods, but nickel / yttrium and cobalt / nickel catalysts are more commonly used.

3.1.2 Laser ablation method:

Laser ablation is an effective method for the preparation of single - walled carbon nanotubes. It is a metal catalyst and graphite mixed graphite target placed in the middle of the quartz tube, the tube is placed in a furnace heating. When the furnace temperature rises to a certain temperature (such as 1473K), through the inert gas, and a laser beam focused on the graphite target. The graphite target generates gaseous carbon under the irradiation of the laser, and the gaseous carbon and the catalyst particles are transported from the high temperature zone to the low temperature region by the gas stream to produce carbon nanotubes under the action of the catalyst.

3.1.3 Chemical Vapor Deposition:

The carbonaceous gas is cracked by a heating or inductance heater, a high temperature furnace or an infrared lamp and a plasma, and the carbon nanotubes are grown on a substrate containing catalyst particles such as Fe, Co and Ni. The method has the advantages of low cost, large output and easy control of the experimental conditions. It is the most promising method for preparing high quality multiwalled carbon nanotubes.

In the later study, it was found that the carbon nanotubes were grown at the top or bottom of the carbon nanotubes, and two kinds of carbon nanotube growth models were proposed: bottom growth and top growth. The free carbon produced by the reaction system dissolves and diffuses in the catalyst particles, and when saturated, the carbon precipitates in the form of tubes. If the adhesion of the catalyst particles to the surface of the substrate is relatively strong, the carbon will precipitate from the upper surface of the particles, i.e., the bottom growth model. If the adhesion of the catalyst particles to the matrix is weak, the carbon will precipitate from the lower surface of the particles and the catalyst particles continue to improve, that is, the top growth model.

3.1.4 Catalytic / reduced pyrolysis in confined systems:

The carbon nanotubes are usually synthesized by catalytic / reducing pyrolytic organic molecules at 400-700 / C using an autoclave as the reaction vessel. This method is simple, easy to operate.

3.2. 3.2 Application of carbon nanotubes [6,11]

3.2.1 Field emission

Carbon nanotubes have been shown to have good field emission properties, and field emission phenomena can be observed at an electric field intensity of less than 1 v / μm, and the current intensity can reach 1A / cm2. According to the nature of carbon nanotubes, micro-arrangement, preparation process and the structure requirements of the transmitter, carbon nanotube field emitters can be made into a variety of structural forms. However, the possible application of single carbon nanotube field emission devices is limited to systems that require very high sharpness to electron sources, such as high resolution electron microscopy and lithographic techniques. In contrast, carbon nanotube film has a great potential for technical applications, it has a friendly launch performance, and easy to prepare and mass production. The field transmitter in the vacuum electronic devices, microwave amplifiers, field emission displays and X-ray tube and other fields has a very broad application prospects.

3.2.2 Sensing

Carbon nanotubes are unique one-dimensional quantum wires and have a very high surface-to-volume ratio, so their electrical properties are very sensitive to molecular adsorption. Especially in semiconducting single-walled carbon nanotubes, all carbon atoms are exposed to the surface so that a very small fraction of the charge generated by molecular adsorption is sufficient to allow the local carrier to run out, resulting in a large conductance change. Based on this, people have built a carbon nanotube-based sensing elements and converters for the detection of gas molecules and biomolecules. Such as H.J.Dai and so on the use of polyethylene imide coated carbon tube developed a highly sensitive, highly selective carbon nanotube sensor. Woollcy et al reported the use of single-walled carbon nanotubes AFM tip directly produced kilobase DNA unit type method, so that people more deeply understand the gene on cancer and heart disease and other common diseases.

3.2.3 Electrochemistry

The diameter of the carbon nanotubes is at the nanometer scale, the spacing between the wall layers is 0.34nm, and the kinetic diameter of the H2 molecules is 0.289nm. Therefore, the hollow cavity and the bundle gap of the single-walled carbon nanotubes are theoretically And interlayer gap can be used as hydrogen absorption position. Therefore, carbon nanotubes hydrogen storage research has aroused widespread concern. A.C.Dllfon and so on using the program temperature desorption method to infer the diameter of 1.2nm, 100% purity 100% single-walled carbon tube hydrogen storage capacity of up to 5-10wt%. Rchen and so on through the multi-walled carbon nanotubes doped with metal Li and K will increase the hydrogen storage capacity to 20wt% and 14wt%, but its chemical stability and hydrogen evolution temperature is still subject to the actual conditions.

4. Fullerenes

The hybridization of carbon atoms in fullerenes is between sp2 and sp3. The earliest discovered and representative structure of the fullerenes is the C60, which is a 20-sided body made up of 20 six-membered rings and 12 five-membered rings. The diameter is 0.7 inm.

Fullerene is a thermodynamically unstable but thermodynamically stable metastable substance in carbon. From the energy point of view, the energy of the carbon atoms in the graphite is zero, and the energy of the carbon atoms in the fullerenes is as high as 0.45eV. Therefore, it is necessary to apply graphite to fullerenes to exert very high energy from the outside.

4.1. Synthesis of fullerenes [11]

4.1.1 Carbon evaporation method:

The carbon atoms are evaporated in the inert gas atmosphere by means of arc, resistance heating, electron beam irradiation, laser evaporation, vacuum heat treatment, plasma and solar energy in the form of artificial / natural graphite or high carbon content coal Ambient pressure and the presence of different types of metal catalysts, the carbon atoms after evaporation are again clustered to form fullerenes.

4.1.2 Catalytic pyrolysis Carbon-containing gases, hydrocarbons and organic compounds:

Fe, Co, Ni and other metals as a catalyst, through the disproportionation of CO, C2H2 or propylene and other gas phase cracking to synthesis. In addition, ferrocene and other organometallic compounds can be directly pyrolysis to produce fullerenes.

4.1.3 Benz flame combustion method:

In the flame temperature 1800K under the conditions of benzene diluted by hydrogen after combustion to C60 and C70, this method is very effective for the preparation of C70, and easy to industrial production.

4.1.4 Conversion of carbon-containing inorganic materials:

At the substrate temperature of 600 / C conditions, by laser irradiation in the crystallization of SiC inside the larger size, less defective fullerenes.

4.2. Application of fullerenes

4.2.1 Superconductor material

The C60 molecule itself is a nonconductive insulator, but when the alkali metal is embedded with the void, the C60 and alkali metal series compounds will be converted to superconductors such as the K3C60 with a high superconducting critical temperature. Compared with the oxide superconductor, C60 series superconductor has a perfect three-dimensional superconductivity, current density, high stability, easy to develop wire and other advantages, is a very valuable new superconducting materials.

4.2.2 New polymer materials

Due to the special cage structure and function of C60, a new functional polymer material with excellent conductivity and optical properties is obtained by introducing the polymer system into a novel functional group. Y.wang reported that the mixture of C60 / C70 was incorporated into the luminescent polymer material of polyvinyl carbazole (PVK) to obtain a new type of polymer photoconductor, its photoconductivity can be comparable with some of the best light guide materials. This kind of light guide material in the electrostatic copy, electrostatic imaging and light detection technology has a wide range of applications.

4.2.3 Other aspects of application

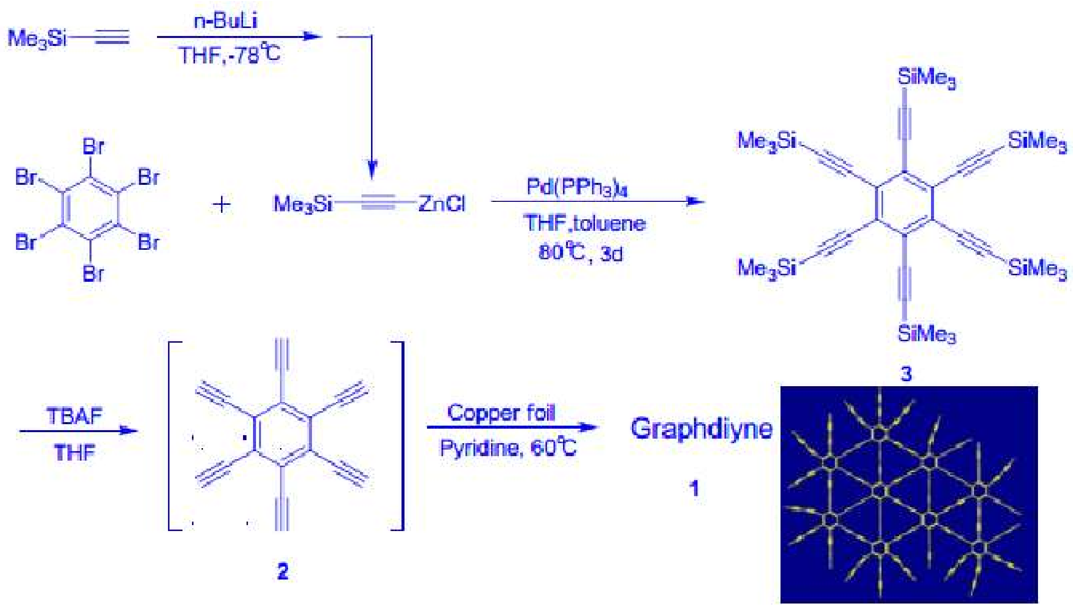
C60F60 as a 'molecular ball' and 'molecular lubricant' in high-tech plays an important role. Lithium atoms embedded in the carbon cage may be made of high-performance lithium battery. Carbon cage embedded in rare earth elements Europium may become a new type of rare earth luminescent materials. Water-soluble C60 derivatives are expected to be used as new nuclear magnetic contrast agents. C60 and its derivatives may become novel catalysts and novel nanoscale molecular wires, molecular pipettes and whisker reinforced composites. The water-soluble host-guest complexes of C60 with cyclodextrin and cyclic aromatics will play an important role in supramolecular chemistry and biomimetic chemistry.

5. Graphite alkyne

Graphite is a kind of carbon isomorphic form formed by sp and sp2 and sp3. The benzene ring is conjugated by 1,3-diyne bond and has a two-dimensional planar network structure. The special structure of the graphite alkyne makes it rich in carbon chemical bonds because the conjugated system is large and has a wide pitch of 4.1913 Å, and the graphite alkyne has good chemical stability and exhibits semiconductor properties.

5.1. Synthesis of graphite alkyne [12]

A wide range of graphene films were successfully synthesized on the surface of copper alloy by hexadecylbenzene catalyzed cross-coupling reaction on copper films. The films were highly ordered, with fewer defects and 10 -3 -10-4 S / m.



5.2. Application of graphite alkyne [13-15]

Du Hailiang, et al. will be doped with graphite acetylene nano-tablets in the organic solar cells within the anode, to enhance the photoelectric conversion efficiency of the battery.

In the field of photocatalysis, WangShuo et al. reported a new type of photocatalyst of graphite-acetylene composite P25, which catalyzes the degradation of methylene blue (MB) by the composite catalyst is superior to the traditional P25 and graphene composite P25 catalyst.

5.2.1 Gas separation

The graphite alkyne has a rich pore structure that makes it an ideal molecular sieve for the realization of various gas separation needs. For example, it can be used as an ultra-thin separation membrane for H2 separation in H2 (mixed between H2, CH4 and CO) -hydrogen

Purified potential application materials.

5.2.2 Desalination

Desalination of seawater using a graphite-based nano-porous membrane is widely considered to be an energy-efficient method that may outperform existing commercial technologies such as reverse osmosis. Buehler et al. found that graphite alkyne voids allow water molecules to permeate without barriers and completely reject salt ions through the carbon 'nanotubes', an excellent feature that is ideal for achieving desalination. Carbon nanotubes can be made from monolayer films of primary graphite alkynes, which are two-dimensional carbon allotropes. Graphite acetylene is highly inert, stable and porous has definite triangular atomic pores and single-atom thick mesh fibers structure.

5.2.3 Hydrogen storage materials

Metal doped graphite alkyne has excellent properties. Because of its additional in-plane π-conjugation that does not possess the sp2 graphene and fullerenes, the graphite alkyne exhibits enhanced binding energy to the calcium, so that they can be optimized for H2 storage materials. Liu and other predicted lithium-modified graphite alkyne can also be used as excellent hydrogen storage materials, the hydrogen storage capacity of up to 18.6%.

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