A New Method for Fabrication and Laser Treatment of Nano-Composites

Hebatalrahman. A
Housing & Building National Research Centre (HBRC), Egypt.
Hebatalrahman@naseej.com

Abstract: A new method for manufacturing of nano-composites was invented; a new technique calls Composite material machine with four strokes was established. Composite material machine is a machine for manufacturing of both plastic and metals matrix composites independent on size, type, and volume fraction of fillers. The machine works in four strokes, each of which worked separately. It depends on the material. The four stokes can be controlled to work in schedule controlled by the main control unit connected to the computer, the machine also work manually. The final products were treated by laser irradiation to improve mechanical properties without any significant change in composition. The new technique is cheap, qualified and simple design. The technique was full automated and has been transferred to industry successfully. [Journal of American Science 2010;6(7):149-154]. (ISSN: 1545-1003).

Keywords: Fabrication, parameters, nanocomposite, laser irradiation, nanoparticles, reinforcement

1. Introduction

Nanocomposites are materials that are created by introducing nanoparticulates (often referred to as filler) into a macroscopic sample material (often referred to as *the matrix*). This is part of the growing field of nanotechnology [1, 2]. After adding nanoparticulates to the matrix material, the resulting nanocomposite may exhibit drastically enhanced properties. For example, adding carbon nanotubes tends to drastically add to the electrical and thermal conductivity. Other kinds of nanoparticulates may result in enhanced optical properties, dielectric properties or mechanical properties such as stiffness and strength [3]. In general, the nano-substance is dispersed into the matrix during processing [3]. The percentage by weight (called *mass fraction*) of the nano-particulates introduced is able to remain very low (on the order of 0.5% to 5%) due to the incredibly high surface area to volume ratio of nanoparticulates. Much research is going into developing more efficient combinations of matrix and filler materials and into better controlling the production process [4, 5].

Historical Background

A. Applications of Nano-composite materials

An important use of nano-particles and nano-tubes is in composites, materials that combine one or more separate components and which are designed to exhibit overall the best properties of each component. This multi-functionality applies not only to mechanical properties, but extends to optical, electrical and magnetic ones [6, 7]. Currently, carbon fibres and bundles of multi-walled CNTs are used in polymers to control or enhance conductivity, with applications such as antistatic packaging. The use of individual CNTs in composites is a potential long-term application. A particular type of nano-composite is where nano-particles act as fillers in a matrix; for example, carbon black used as a filler to reinforce car tyres. However, particles of carbon black can range from tens to hundreds of nanometres in size, so not all carbon black falls within our definition of nano-particles [8-10].

b. Laser Method

In 1996 CNTs were first synthesized using a dual-pulsed laser and achieved yields of >70wt% purity. Samples were prepared by laser vaporization of graphite rods with a 50:50 catalyst mixture of Cobalt and Nickel at 1200°C in flowing argon, followed by heat treatment in a vacuum at 1000°C to remove the C60 and other fullerenes. The initial laser vaporization pulse was followed by a second pulse to vaporize the target more uniformly [11]. The use of two successive laser pulses minimizes the amount of carbon deposited as soot. The second laser pulse breaks up the larger particles ablated by the first one, and feeds them into the growing nano-tube structure. The material produced by this method appears as a mat of "ropes", 10-20nm in diameter and up to 100µm or more in length. Each rope is found to consist primarily of a bundle of single walled nano-tubes, aligned along a common axis. By varying the growth temperature, the catalyst composition, and other process parameters, the average nano-tube diameter and size distribution can be varied. Arc-discharge and laser vaporization are currently the principal methods for obtaining small quantities of high quality CNTs. However, both methods suffer from drawbacks. The first is that both methods involve evaporating the carbon source, so it has been unclear how to scale up production to the industrial level using these approaches. The second issue relates to the fact that vaporization methods grow CNTs in highly tangled forms, mixed with unwanted forms of carbon and/or metal species. The CNTs thus produced are difficult to purify, manipulate, and assemble for building nanotube-device architectures for practical applications[12-14]

2. Material and Methods

Experimental study

a) The System Descriptions

When the four stroke composite material machine is describe, it is not only machine for manufacturing of composites but it is a complete system for manufacturing include grinding of raw materials, addition of additive, mixing and manufacturing in four steps. The manufacturing parameters are indicated as temperature, Pressure, cooling rate, and time of application of force. Fig (1) shows the main steps of manufacturing.

The unit Dimensions =Height 4.5m-* width 5m *length 7m

The machine operations follow the following steps:-

1-Grinding to prepare raw materials in nano-size

2-The nano-size particles moves on the belt to add additive and other reinforcement

3-All components was mixed in a multi-speed mixer

4-The powder mixture was added to the iron mold

5-The mold was heated and treated with lubrication before mixture added

6-The manufacturing cycle work in four steps heating, Pressing, cooling and extraction of final products

7-The electronic arm moves mold and mixture away from heaters to the cooling path and vise verse

8- The control unit was adjusted to control both manufacturing parameters and movement of the electronic arm.

Fig (2) shows the workshop design and Fig (3) shows the main parts of the four stroke composite material system. The system design was simple and abundant to be suitable for small investment and youth projects. The system is suitable for plastic matrix composite, metal matrix composite, powder metallurgy, recycling of powder waste and fabrication of thermoplastics and thermosets.

b) Fabrication of composite

To prove the quality of the system, it was used to prepare FRP (plastic matrix composite) PMMA reinforced by fiber glass. PMMA (poly methyl metha acrylate) are Considered as the material which have the best biocompatibility to be used in medical applications, but have a lot of disadvantages such as low hardness, low strength, high shrinkage, high water absorption and low thermal conductivity. These disadvantages have already overcome by making composite material consists of PMMA (poly methyl metha acrylate) as matrix and use fibre glass type E as reinforcement. Type E fibre glass has a lot of characteristics because it is cheap, abundant and effective to overcome the disadvantages. Fibre glass was used in Volume fractions (zero %, 17 %, 35%, 50%, 65%) and the length to diameter ratio were (L/D = 1) (L/D=50). The manufacturing quality parameters are indicated as temperature, pressure, time of application of force and cooling rate. The material is treated using two different types of lasers such as Argon — ion laser with wavelength 514. 5nm and Nd — YAG with 355nmwavelength(third harmonic generation).

C) Material Preparation

The material prepared by grinding and mixing for PMMA in the powder form which have diameter less than 100µm with short fibre type E-fibre glass which is mechanically cut to certain sizes. The fibre size which is recorded by length to diameter ratio L/d=50 Ownes Corning Co, 731ED 1/32" was chosen. The mixing of fibre with PMMA is done with certain volume fractions as mentioned above.

d) Determination of Manufacturing Parameters

The manufacturing parameters were adjusted and the effect of temperature, pressure and cooling rates were recorded. Design and test of the new system were evaluated. The nano- composite material consists of poly methyl metha acrylate as matrix reinforced with fibre glasses as filler The manufacturing steps may be summarized in the following steps:-

1-Grinding of PMMA by electrical blinder-and control the grinding time to be less than 1.5 min to avoid temperature rise and agglomeration of particles after that use mesh < 300µm diameter for sieving.

2-Short fibre have used in powder form.

3- Mixing of the two powders.

4- Heating the mold.

5- Compress the mixture with (5:10 ton) hydraulic system.

6- Cooling the mold with water JET.

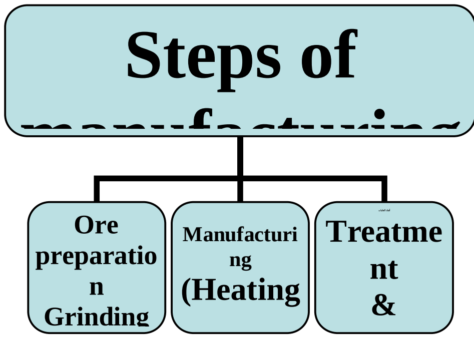


Fig (1) Steps of Manufacturing

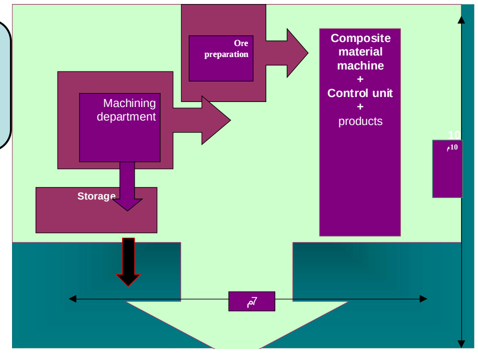
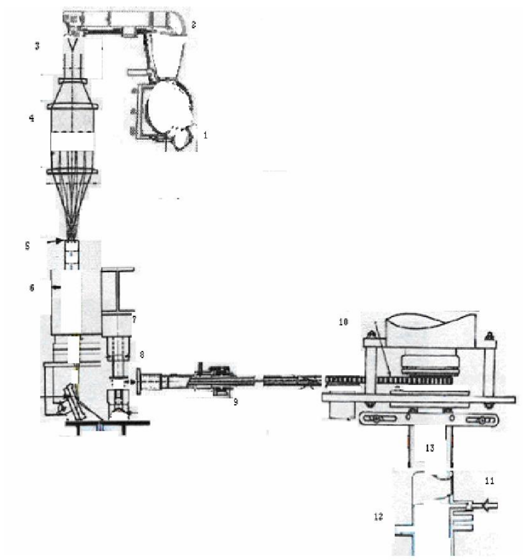


Fig (2) The workshop design



(Four Stroke Composite Material Machine)

1-Grinding machine
2-belts
3-addition of raw materials
4-multi speed mixer
5-Cones for mold feeding
6- Shaping mold
7-press 100ton
8-Heaters 220volt,3000watt, 500C
9- electronic arms
10-control of automatic arm
11- water inlet
12-water outlet
13- fans

Fig (3) Main parts of the four stroke composite material system

e) Laser Irradiation

Samples used in this investigation were in a shape of disk of 25mm diameter and 7mm length. The irradiation is done on both sides of the samples and in different positions to cover all the area of the sample and achieve homogeneous surface suitable for testing .The different lasers irradiated samples at different conditions were examined before and after laser irradiation and the effect of laser was determined.

The type of laser used is Nd-YAG I used in the third harmonic (wavelength =355nm) which lies in the ultra violet (UV) region with power of bout 10milli watt, duration time 7nsec,repettion rate 17HZ and energy (40mmj/pulse) [6]

F) Microscopic Examination

All specimens were prepared metallographically first by grinding on different grades of silicon carbide “SiC” paper coarse grinding and then fine grinding. The process had to be repeated several times to obtain best results and to produce uniform level of matrix. All of these pictures were carried out in several areas in each specimen by making negative at 500x by Olympus optical microscope

g) Raman Spectroscopy

The behaviour of the material was studied by variation of the modes obtained from FT (IR) Raman spectrum. The results were obtained using Brucker FTIR Raman spectrometer using Nd-YAG laser power of 500MW as source of excitation. After irradiation the thin film of sample with different lasers, the spectra were obtained in the range 400 to 4000 cm-1. The samples to be measured was fixed to holder and placed in front of the beam [7].

3. Results and Discussion

Fabrication Procedures

The fabrication quality parameters are indicated as follow: -

1. Maximum fibre percentages to achieve success fabrication were (65%) increasing the fibre percentage up on this value will produce less qualified fabricated material.

2- Temperature is the most significant parameter in Fabrication. Over heating temperatures at all fibre percentage leads to dissociation of PMMA to monomers and different gases. As a result of low fabrication temperatures, viscosity becomes insufficient for deformation so the optimal deformation temperature is required to get viscous matrix suitable for addition of reinforcement.

3- The pressure required for deformation and time of force act were strong functions of both volume fraction and fibre percentages.

5- The fast cooling rate (water cool) may be recommended as the suitable Cooling rate regardless of fibre percentage and volume fraction.

6- The density of nano-composites will not be affected by laser irradiation.

7- Laser irradiation is used to improve the surface properties of the material as follow:

A - The hardness will improved from (43 to 63) barcoul about (50%) when the exposure time increase from 21 nano second to 70 nano second and after that the hardness remain constant with increasing time.

B — the hardness improved with increasing fibre percentages and the maximum value was (60 Barcoul) at L/D = 1 and volume fraction 50%. This improvement increase with laser irradiation and reach its maximum at (80 Barcoul) with argon - ion laser at L/D =50 and volume fraction 50%.

C — the wear rate is improved with fibre percentage to reach (3.8m/m) at L/D = 50 and volume fraction 50%.

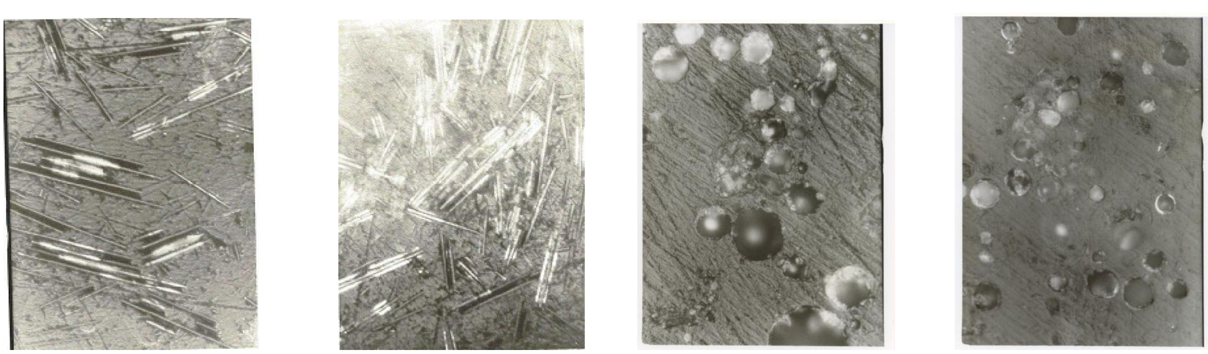
8 – The wear rate and coefficient of friction were improved more with laser irradiation to reach 0.4m/m wear rate and 0.75 coefficient of fraction at argon irradiation (L/D =50 and volume fraction 50%)

9- Alkali resistance is excellent before and after laser irradiation and weight loss is less than 0.01% at exposure time more than 30000 hours.

All of the above tests were done according to standard methods ASTM [3-7]

b) Microstructure Analysis

The reinforcement strength to weight ratio and volume fraction have a significant effect on the manufacturing conditions and mechanical properties. Fig (4-a) and fig (4-b) show the microstructure of nano-composites when the length to diameter ratio equal 50, the fiber volume fraction is 17% and 50% respectively, fibers were distributed in random manner in the structure and cross linked together in different positions, this distribution prevents the crack propagation. In other words, the crack does not reach the critical size. Cross linking of reinforcement in the matrix is the main reason of improvement in the mechanical properties. When the volume fraction of fibers increased to 50%, the cross linking of fibers were increased, the crack propagation became very limited. The mechanical properties were improved as a result of increase in fiber volume fraction. In fig (4-c) and fig (4-d) show the microstructure of nano-composites when the length to diameter ratio equal 1, the fiber volume fraction is 17% and 50% respectively. When the fiber shape was circular the cross linking was relatively limited, when compared with the fibers in fig (4-a), that is the main reason for extra improvement in mechanical properties when the fiber size (length to diameter ratio) increased at the same volume fraction. When comparing fig (4-d) with both figure (4-a) and (4-b), the random distribution of particles with high volume fraction 50% improves the mechanical properties and crack propagation theme better than fig (4-c). The amount of cross linking was still lower than both fig (4-a) and fig (4-b). The qualitative analysis of the microstructure of nano-composite explains the improvement in mechanical properties in terms of fiber shape, size and volume fraction. The laser irradiation effect was not detected by optical microscope so spectroscopic analyses of the samples were done.



A) 17% fibre, L/D=50, 500x b) 50 % fibre, L/D=50, 500x C) 17% fibre, L/D=1, 500x D) 50 % fibre, L/D=1, 500x

Fig (4) the fibre impeded into the matrix before laser irradiation at 500x and at different percentages of fibres.

c) Raman Spectroscopy

Laser irradiation was a very good method for treatment of nano-composite without any change in morphology of the samples surface. Fig (5) Raman spectrum of the nano-composite at different length to diameter ratio and different volume fractions before and after laser irradiation by Nd-YAG and Argon lasers. The spectrum was affect by laser type volume fraction of fiber, fiber shape and length to diameter ratio (fiber size). In quantitative analysis of the IR spectrum of the material each absorption band is characterized by four parameters

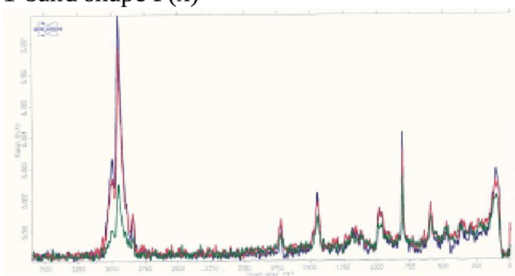
1-band shape F(x)

2-Intensity of the band (integral intensity rather than peak depth)

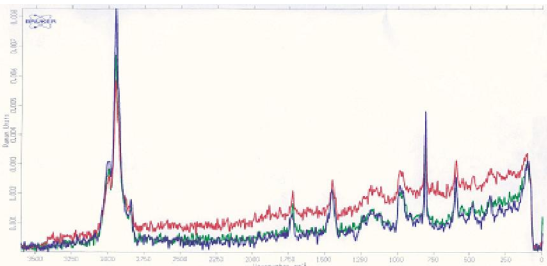
3-Half band width of the band (the width of the band at its half depth)

4-The centre of mass of the band.

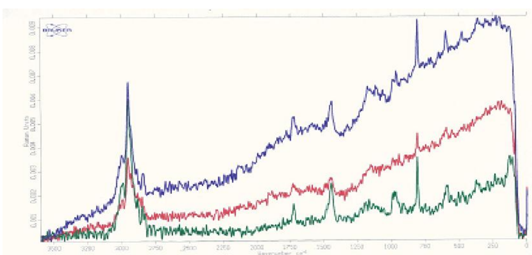
The complex behaviour reflecting two regions of dependence on the characteristics of lasers, which might be related to the structure changes and introduce the parameters characterizing the vibration motion of the vibrating groups such as the vibration activation energy in certain vibration energy levels.



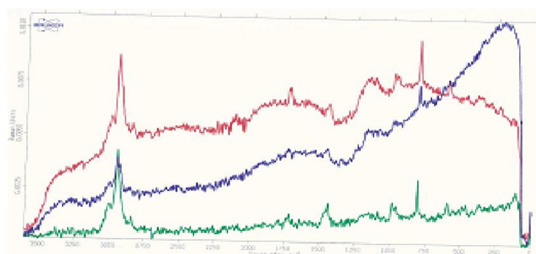
a) L/D=1, VF=17%



b) L/D=50, VF=17%,

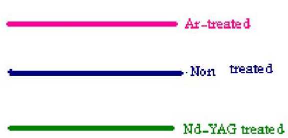


VF=50% ,L/D=50 d)



% VF=50 c) L/D=1,

Fig (5) Raman spectrum of the nano-composite at different length to diameter ratio and different volume fractions before and after laser irradiation by Nd-YAG and Argon lasers.



Fiber is considered as the element that more affected by laser in the structure, the change in fibre shape leads to increase in the area exposed to lasers. Argon laser treated samples recorded higher peak values almost at all curves. The results were relatively compatible with the mechanical properties. The ability of material to absorb a certain laser wavelength is a property of the material as a function of laser type, wavelength, power and repetition rate.

4. Conclusions and Recommendations

Four strokes composite material machine is considered as economic and qualified method in production of nano-composites. The system is a multifunction system in compact design.

The initiative produces scientific contribution in a very specific field. The production of thermoplastic based composite material is a new trend all over the world, with these patents Egypt is one of the countries all over the world build and transfer new technology, the initiative produces 40 compounds contribute in more than 120 industries. Now shafts, gears, rods, chairs, tables, and electrical equipment are made from composites. Very small units for production of the parts mentioned above is established, this small unit works full automatic

The following Recommendation may be considered for future research:

1- Irradiation of fiber glass before manufacturing

2- Using laser in cutting of fiberglass and study how the fiber properties changes.

3- Using different types of lasers with different wavelength and study the differences.

4- Study the effect of laser irradiation on other types of fiber and matrix to reach the optimum properties and best treatment method.

5- Study the different manufacturing parameters to increase the economical value.

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