EFFECT OF BALL MILLING ON MECHANICAL PROPERTIES OF CARBON NANO TUBES AND GRAPHENE REINFORCED WITH ALUMINIUM COMPOSITES

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ABSTRACT:

In recent years Carbon nanotubes and graphene has attracted considerable research interest in all fields of nano materials due to its unique properties. Its excellent mechanical properties lead it to be used in nano-composites for strength enhancement. In this investigation, carbon nanotubes (CNTs) and Graphene reinforced aluminium composites were prepared by using ball milling. Ball milling leads to the proper intermolecular level of mixing, that leads to breaking of strong intermolecular bonds between the particles and leading to the good results. The composites were prepared by 2% constant weight percentage of CNTs and varying weight percentage of graphene(0.5% and 1%) with aluminium for different ball milling speeds. Scanning electron microscopy, X-ray diffraction, were carried out to characterization of nano composites. The effect of ball milling on hardness, density properties of composites was investigated. Experimental results showed that nanotubes and graphene were homogenously distributed in 0.5% wt of composites but for 1% wt leads to agglomeration of the composites. It was observed that the hardness and porosity of the hybrid composite increased with increasing reinforcement volume fraction and density decreased with increasing particle content.

Keywords: Carbon nanotubes, Graphene, Aluminium, Fabrication, Mechanical properties.

1. Introduction

Nanotechnology is the art and science of manipulating matter at the nano scale to create new and unique materials and products with enormous potential to change society. Particle with nominal diameter of 100 nano meter in nature (smoke, dust) human expose and accommodation nothing new, we can't assume that nano materials are the same as their bulk counterpart. But also can't assume that they are more toxic. Every particle should be tested on case by case basis. Nano materials have properties that are significantly different and considerably improved relative to those of their coarser-grained counterparts. The property changes result from their small grain sizes, the large percentage of their atoms in large grain boundary environments and the interaction between the grains. Research on a variety of chemical, mechanical and physical properties is beginning to yield a glimmer of understanding of just how this interplay manifests itself in the properties of these new materials. In general, one can have nano particles of metals, semiconductors, dielectrics, magnetic materials, polymers or other organic compounds. Semiconductor hetero structures are usually referred to as one-dimensional artificially structured materials composed of layers of different phases/compositions. The semiconductor hetero structured material is the optimum candidate for fabricating electronic and photonic nano devices. The term nano originated from the Greek nano which means 'dwarf'. It is one billionth of a meter. Therefore, whenever we think about nano science or nanotechnology, very small objects come to the mind. Indeed, this branch of science and technology deals with materials having at least one spatial dimension in the size range of 1 to 100 nm[1].

Carbon nanotubes are near ideal whisker consisting of seamless cylinders formed by mixing graphene and exhibit the many remarkable Mechanical, electrical and Thermal properties. Depending on their length and diameter, properties are varied. CNT shows almost five time's elastic modulus (1TPa) and closely 100 times the tensile strength (150GPa) than the high strength steels. CNTs are light weight regards as nano-reinforcement for composites are quite promising [2].

Graphene has attracted considerable attention in the last several years because of properties such as high mechanical strength and modulus, electrical and thermal conductivity and optical transmittance. Fabrication methods have been devised to create single layer and multilayer graphene and graphene oxide in small quantities, with the intent to find methods that will result in bulk quantities of graphene for use in applications such as composites. There have been a limited amount of studies on the behaviour of graphene composites. While studies have primarily been concerned with enhancing the properties of polymer matrices, with some results having shown great promise, there has been little to no research in metal matrices. This is likely a result of the greater difficulties in dispersion and fabrication, and the unknown inter-facial chemical reactions in metal composites. This disparity in the amount of research given to polymer matrices as compared to metal matrices is seen in carbon nanotube composites as well [3].

Aluminium has been a common material to study in metal–carbon nanotube composites due to the diverse range of technical applications for lightweight alloys. Researchers have seen mixed results with some reporting little or no increase in mechanical strength. While others have seen significant increases in strength. Many of these differences are a result of the quality of dispersion, fabrication method, and interfacial reactions that occur. In this study, graphene platelets derived from graphite oxide are combined with aluminium in order to observe the effects on mechanical strength. Very limited research has been done in the field of carbon nanotube reinforced metal matrix composites due to the factors that uniform dispersion of carbon nanotubes in metal matrix is quite difficult; the interfacial reaction between carbon nanotubes and metal matrix may be rather serious resulting in the deterioration of composite properties, and the suitable fabrication technique also is very lack. As metal powder size is much larger than that of carbon nanotubes, it is difficult to achieve homogeneous distribution of carbon nanotubes in the composites [4-5].

Noguchi et al. reported a nano-scale dispersion method in CNT/Al composites by introducing into an elastomeric precursor. Cha et al. found a molecular level mixing method in CNT/Cu composites by means of a salt containing Cu ions. Furthermore, Hu et al. showed an in situ reduction approach in CNT/Au nanoparticles composites materials. In order to obtain excellent mechanical and physical properties, Dong et al. reported an improvement on fracture toughness, wear resistance and hardness in CNT/Cu composites fabricated by hot pressing sintering. Laha et al. reported that a 71.8% increase in micro hardness in CNT/6061Al composites by plasma spray forming when 10 wt. % MWNTs was added to Al matrix. Tang et al. found a 65% decrease in coefficient of thermal expansion (CTE) when 15 vol. % carbon nanotubes were added to the nano-Al matrix. Moreover other researches on carbon nanotube/metal matrix composites reported improvements in mechanical properties when appropriate carbon nanotubes content was added [6-12].

2. MATERIALS AND METHODS

2.1 Material Used

Aluminium has about one-third the density and stiffness of steel. It is easily machined, cast, drawn and extruded. Corrosion resistance can be excellent due to a thin surface layer of aluminium oxide that forms when the metal is exposed to air, effectively preventing further oxidation. Carbon Nanotubes are the strongest materials used in any aspects, it has been used in almost all the stressed parts in automobiles, aircrafts and ship building etc. Graphene is one kind of material which has the strongest binding and stressful conditions to use in heavy weight carrying and wear conditions. Key Properties of this material are like, it is having medium to high strength., good toughness., good surface finish., excellent corrosion resistance to atmospheric conditions., good workability, widely available.

2.2 Reinforcements

2.2.1 Carbon Nanotubes

Carbon Nanotubes are allotropes of carbon with cylindrical nano structure. This cylindrical carbon molecules have unusual properties, which are valuable for Nano technology, electronics, optics and other fields of material science and technology. Some of the properties are shown in Table 1.

Table 1: Properties of CNT's

Properties	Specific Density (kg/m^3)	Young's Modulus (Tpa)	Strength (GPa)	Strain of Break (%)	Thermal Conductivity (W)	Electrical Conductivity(s/m)
Values	1.3 – 2	1	10 – 60	10	>3000	10^6 - 10^7

2.2.2 Graphene

Graphene (fig.1) is an allotrope form of carbon consisting of a single layer of carbon atoms arranged in a hexagonal lattice. Some of the key properties are, most reactive form of Carbon, tensile strength exceeds 1 Tpa and it is stretchable up to 20 of its initial length. Some of the properties are shown in table 2.

Table 2: Properties of Graphene

Properties	Stiffness	Strength	Toughness
Values	1 Tpa	42 N/m	4 – 4.6 MPa

Fig 1.: Graphene

Fig 2: CNT's First sample is pure

aluminium (97.5%), fixed weight percentage CNT (2%), graphene (0.5%) , Aluminium(97%) , CNTs(2%) and graphene (1%) samples composition by varying ball milling time 0.5hr, 1hr,1.5hr, ball milling process in the planetary ball milling machine with (1:10) ball to powder ratio. Ball milling speed is maintained at 250 rpm and small amount of acetone is used to reduce the heat produced in the process. After bal milling the powders were compacted using Universal testing Machine with a load range 75KN to 90KN, the samples are shown in fig 3.

Fig 3: specimens after sintering

2.3 Preparation of powder samples

In the case of fabrication of CNTs-MMCs through powder metallurgy process, Al fine powder as matrix and fixed weight percentage (2%) MWCNTs having diameter 30-50nm, graphene 30nm thickness (varying percentage of 0.5% and 1%) were used as reinforcements. Planetary ball milling used to blend the powders by varying ball milling time 0.5hr, 1hr and 1.5hr with 250rpm speed constant for samples. The weight percentage and time variation of each sample is shown in the below Table.3&4.

Table 3: Weight ratio of powder samples

Sample Number	Al	CNT	Graphene
S-1	100%	0%	0%
S-2.1	97.5%	2%	0.5%
S-3.1	97%	2%	1%

Table 4: Speed and time of milling sample

Sample number	Speed in rpm	Time in min
S-1	300	60
S-2.1	300	30
S-2.2	300	60
S-2.3	300	90
S-3.1	300	30

S-3.2	300	60
S-3.3	300	90

3. EXPERIMENTATIONS

3.1 Compacting and Sintering

After ball milling the powder samples as shown in fig.4, the powder samples were compacted by the using die as per ASTM standards. After compaction, the green samples are as shown in fig .5. The green samples were processed for sintering carried out with temperature 580°C, constant for all the samples. Sintering is done for 8hours nitrogen atmosphere to prevent oxidation of the specimens.

Fig.4: Powder samples

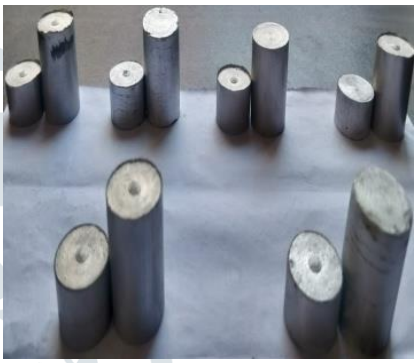

Fig.5:Green samples

3.2 Density Test and Hardness Test

A material's density is defined as its mass per unit volume. In other ways, density is the ratio between mass and volume or mass per unit volume. Density is essentially measurement of how tightly matter is crammed together. The principle of density was discovered by the Greek scientist Archimedes, but it is easy to calculate if u know the formula and understand its related units. By the formula below the Actual density can be calculated to compare the values between reinforced and non-reinforced materials given by Equation -1, Fig.6 shows the density testing apparatus.

$$Density = \frac{W(air)}{[W(air) - W(water)]} \ldots Eq\ (1)$$

Hardness is defined as the ability of a material to resist plastic deformation. Micro Hardness testing machine as shown in fig.7 determines the degree of deformation of a material and it is generally accepted as an important property and a valuable parameter of comparison with the tooth structure. We are using micro hardness testing machine which is more accurate and easier to measure the indentation than Vickers and Brinell. Indenter used is Diamond indenter. It is having a magnification of 100X and 500X.

Fig6: Density testing apparatus

Fig 7: Computerized Micro Hardness Testing Machine

3.4 Scanning Electron Microscope

A Scanning Electron Microscope (SEM) as shown in fig.8, is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the surface topography and composition of the sample. The electron beam is scanned in a raster scan pattern, and the position of the beam is combined with the intensity of the detected signal to produce an image. In a typical SEM, an electron beam is emitted from an electron gun fitted with a tungsten filament cathode. Tungsten is normally used in thermionic electron guns because it has the highest melting point and lowest vapour pressure of all metals, thereby allowing it to be electrically heated for electron emission, and because of its low cost. Other types of electron emitters include lanthanum hex boride (LaB6) cathodes, which can be used in a standard tungsten filament SEM if the vacuum system is upgraded or field emission guns (FEG), which may be of the cold-cathode type using tungsten single crystal emitters or the thermally assisted Schottky type, that use emitters of zirconium oxide. For study of dispersion of the reinforcements in the matrix.

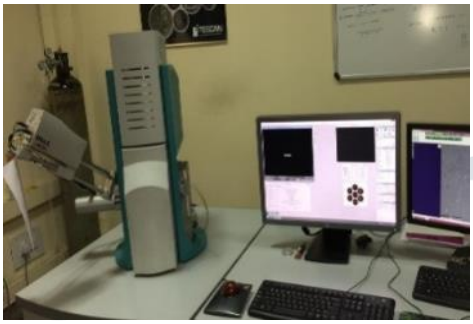

Fig.8: Scanning Electron Microscope

3. Result and discussion

3.1 Morphology of the powder

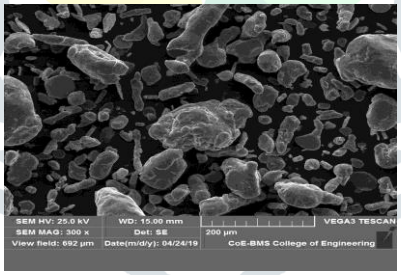

Fig 9.1: SEM image of S-1

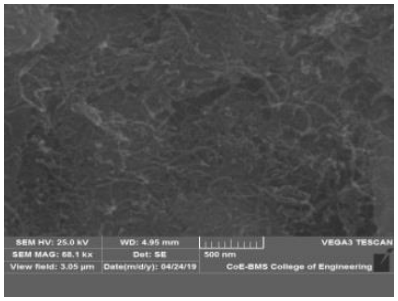

Fig 9.2 : SEM image of S- 2.1

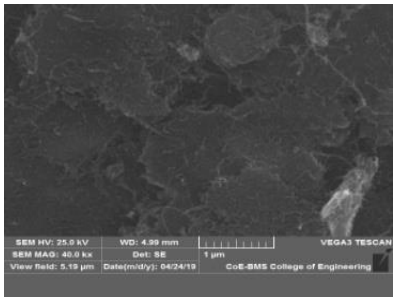

Fig 9.3 : SEM image of S- 2.2

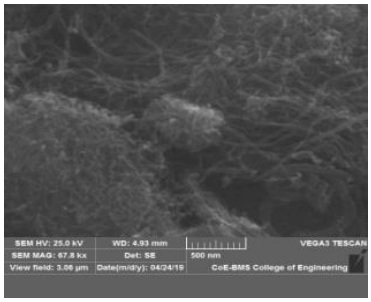

Fig 9.4 : SEM image of S- 2.3

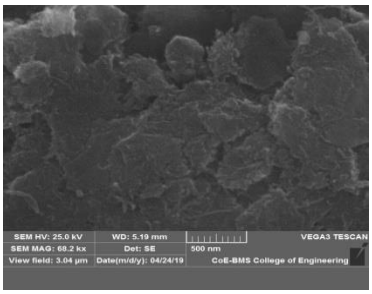

Fig 9.5 : SEM image of S- 3.1

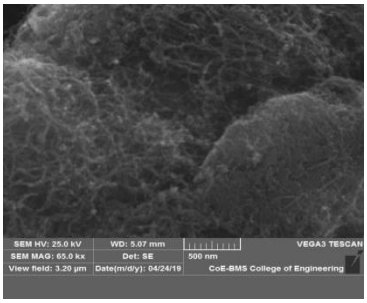

Fig 9.6: SEM image of S- 3.2

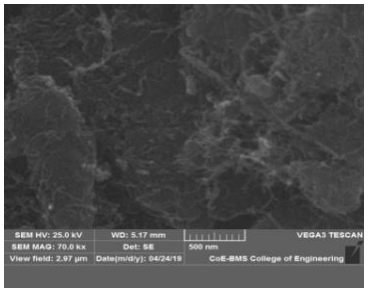

Fig 9.7: SEM image of S- 3.3

Fig 9: SEM images of Nano composites

Above all SEM images representing the morphology of the samples. First samples is pure Aluminium and other samples are nano hybrid samples. All images are shown above are SEM images of powdered samples. Fig 9.2 to 9.4 representing the SEM image of Aluminium and 2% CNT and 0.5% graphene, it reveals uniform distribution of reinforcement for increasing ball milling time . Fig 9.5 to fig 9.7 SEM image of Aluminium and 2% CNTs, 1% of graphene, the results shows that 1% graphene with 2% of CNTs are difficult to reinforcement, leads agglomeration.

In all the images it is evident that carbon nanotubes and graphene are tangled together. It can be found that Aluminium powder mixes with CNT and graphene still keeps near sphere shapes with size about 1-100 micro meters.

3.2. Density and hardness properties of composites

The effect of carbon nano tubes content on relative density and hardness are discussed below. It is evident that with small amount of CNTs and graphene additions, has effect on all the parameters of the mechanical properties such as relative density and hardness. Large amount of CNTs and graphene may reduce the properties. small amount of CNTs and graphene can fill micro voids of aluminium resulting in the increases of density, hardness and few other parameters.

CNTs agglomeration not only impends the densification of the specimens, but also become the defect source. Hence the relative density and hardness of the composites decreases.

Table.5 Density test

Sample number	Theoretical density (g/cm^3)	Actual density (g/cm^3) After sintering
S-1	2.7	2.56
S-2.1	2.685	2.59
S-2.2	2.685	2.53
S-2.3	2.685	2.59
S-3.1	2.683	2.53
S-3.2	2.683	2.57
S-3.3	2.683	2.59

Hardness is defined as the ability of a material to resist plastic deformation .Hardness determines the degree of deformation of a material and it is generally accepted as an important property and a valuable parameter of comparison with the tooth structure. increase in the ball milling increases, since uniform distribution of reinforcement. For 1% wt nano Graphene percentage composites is more difficult to distribute in between matrix. The below fig.9 gives the information about the results obtained from hardness test.

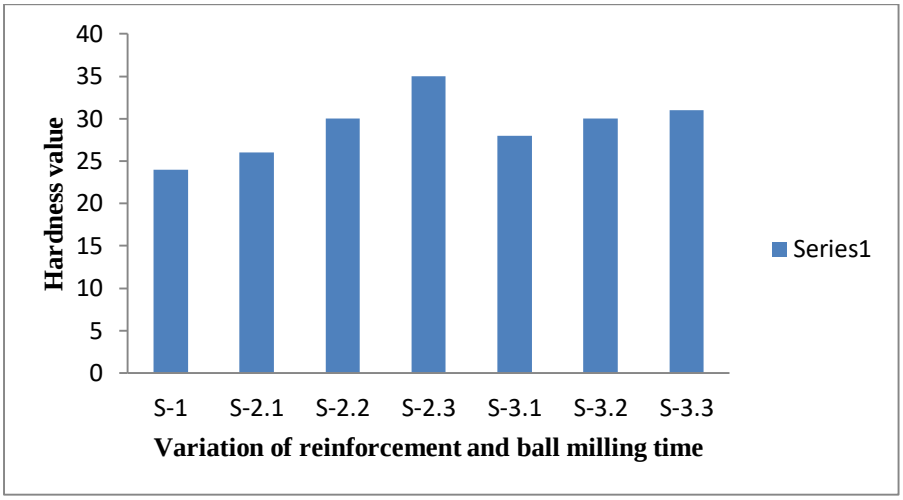

Fig.10 Hardness test

The Above fig10 represents that microhardness test results. hardness increases with increasing reinforcement and ball milling time but for 2% CNTs and graphene hardness increases not much due to more weight percentage of nano reinforcement in matrix.

4. Conclusion

- Powder samples were successfully ball milled for varying ball milling time and nano composites samples were fabricated using powder metallurgy technique.
- actual density of composites is increases with increase in milling time whereas theoretical density decreases
- Hardness increases with increases in Graphene and CNTs reinforcement and also increases with increasing ball milling time.
- Carbon nanotubes are successfully retained, dispersed uniformly in the composites for 0.5% graphene but 1% graphene shows agglomeration.
- Theoretical density of composites is decreases with increase in reinforcement but actual density increases with milling time and reinforcement.

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