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Determination of Mechanical Behaviour of Fe-CNT MMC*

C. Parswajinan[a,*], B. Vijaya Ramnath[b], M.Vetrivel[c], A. Ranjith[d], B. K. Rohit[e], S. Vasanth[f] and S. Vignesh[g]

[a]*Research Scholar, Department of Mechanical Engineering, SCSVMV University, Kancheepuram, Tamil Nadu – 631561, India and Asst. Prof., Department of Mechanical Engineering, Sri Sai Ram Engineering College, Chennai, Tamil nadu – 600044, India*

[b] *Professor, Department of Mechanical Engineering, Sri Sai Ram Engineering College, Chennai, Tamil nadu – 600044, India*

[c]*Professor, Department of Mechanical Engineering, SCSVMV University, Kancheepuram, Tamil Nadu – 631561, India*

[d, e, f, g]*Department of Mechanical Engineering, Sri Sai Ram Engineering College, Chennai, Tamil nadu – 600044, India*

Abstract

Composite Material have found widespread application in various fields. Carbon Nanotubes have recently emerged as materials with outstanding properties. However, until now, the main obstacle is to obtain a homogenous dispersion of the CNTs in the desired material matrix. The manufacturing of Metal/CNT specimen is done by Powder Metallurgical process, advanced technology of manufacturing over the conventional manufacturing process with less cost, time saving and reduced secondary operation. In this project iron metal powder of fine mesh was used and MWCNT was used as a reinforcement as indeed. The reinforcement is done in various composition. The iron powder and CNT, with liquid paraffin used as a binder, is blended by ball milling process. Then the powder was compacted in hydraulic press. The compacted specimen, called Green specimen, was sintered with Nitrogen atmosphere. The sintered component with various composition of CNT is fabricated. The composites made out of a metal and CNT are characterized regarding microstructure and Mechanical properties.

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Keywords: MMC; CNT; Die; Powder Metallurgy

1. Introduction

A material in which a continuous metallic phase (the matrix) is combined with another phase (the reinforcement) that constitutes a few percent to around 50% of the material's total volume. In the strictest sense, metal matrix composite materials are not produced by conventional alloying. This feature differentiates most metal matrix composites from many other multiphase metallic materials, such as pearlitic steels or hypereutectic. Multi-walled carbon nanotubes (MWNTs) reinforced Si coatings using solid reaction method between pure silicon powders and carbon nanotubes (CNTs) was prepared. CNTs reinforced AZ91 metal matrix composites (MMC) were fabricated by the squeeze infiltrated method. The squeeze infiltration technique was a proper method to fabricate magnesium matrix composites reducing casting defects such as pores and matrix/reinforcement interface separation etc. Improved tensile

* Corresponding author. Tel.: +91-984-049-4561;

**E-mail address:* parswajinan@gmail.com

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strength was obtained by reinforcing Si coated CNT to magnesium alloys [1]. CNTs reinforced porous CuSn oil bearings was prepared by powder metallurgy method Surfaced modification of CNTs is made to ensure interfacial bonding between CNTs and CuSn matrix. The results reveal that coated CNTs can improve mechanical properties and oil content of the porous CuSn oil bearings if they are evenly dispersed. Adding too much CNTs will degrade microstructure of the porous bearings because they are difficult to disperse. It will bring negative effect on the mechanical properties and oil content of the porous bearings [2]. Semi-solid powder processing (SPP) is a promising technology that combines the benefits of semi-solid forming and powder metallurgy. Carbon nanotube (CNT) reinforced aluminum alloy 6061 (Al6061) composite was synthesized by SPP for the first time. Microstructure and the fracture surface analyses showed that the CNTs were uniformly dispersed throughout the Al6061 Matrix. Higher density composite was obtained at higher liquid content, although the highest composite hardness was achieved when processed at 620 ◦C. It was speculated that the formation of carbides at higher temperatures affected the interface bonding between the matrix and CNT. The study showed feasibility of manufacturing CNT reinforced metal composites by SPP [3]. Nanocomposite were prepared via mechanical alloying (MA) of Al powders with single-, double, and multi-walled carbon nanotubes (SWCNTs, DWCNTs, and MWCNTs) as reinforcements in order to investigate the MA behaviour and resulting mechanical properties depending on the types of the CNTs. Consequently, only in the MWCNT–Al nanocomposite samples, significant improvement of mechanical property as compared to sintered pure Al samples was observed. Possible origin of the iamprovements in mechanical properties, despite the predicted poor CNT–Al interfacial bonding, was proposed conceptually [4]. The mechanical properties of extruded Ti/CNTs composites at elevated temperature were remarkably improved by adding a small amount of CNTs, compared to extruded Ti matrix [5]. It has been found that the mechanical properties of Al-f CNT composites were much superior to the composites fabricated using non-functionalized or acid functionalized carbon nanotubes [6]. Magnesium containing 6 wt.% aluminum alloy composites reinforced with carbon nanotubes were fabricated with powder metallurgy based wet-processing. Field emission-transmission electron microscopy microstructural analysis clarified that needle-like ternary carbides of Al2MgC2 were synthesized at some interfaces between magnesium matrix and carbon nanotubes, and the other interfaces were clean without any other materials or defects [7]. Carbon nanotube (CNT) composites are fabricated by direct in-situ growth of CNTs on the Zirconia particles, followed by densification via the Spark Plasma Sintering (SPS) technique. Scanning electron microscopy analysis of the 3YTZP-CNT powders shows uniform distribution of CNTs without the formation of agglomerates frequently seen with the traditional ex-situ mixing of CNTs in ceramic compositions. The samples were sintered to nearly 100% theoretical density and with a finer grain size microstructure [8]. carbon nanotube can improve the mechanical properties of highly-conductive low strength copper metals, whereas in low-conductivity high-strength copper alloys the electrical conductivity can be improved [9]. The large aspect ratio CNTs used in the present study were difficult to disperse at CNT wt.% greater than 2, and thus the expected improvements in mechanical properties with increase in CNT weight content were not fully realized [10]. A novel particles-compositing method was used for the first time to disperse different contents of multiwalled carbon nanotubes (CNTs) in micron sized copper powders, which were subsequently consolidated into CNT/Cu composites by spark plasma sintering (SPS). The composite containing 15 vol.% CNTs led to a rather low thermal conductivity due possibly to the combined effect of unfavourable factors induced by the presence of CNT clusters, i.e. large porosity, lower effective conductivity of CNT clusters themselves and reduction of SPS cleaning effect [11]. The chips with the well dispersed MWNTs on their surface were melted and at the same time vigorously stirred. The compression at failure, the compressive yield strength and ultimate compressive strength have all been improved significantly up to 36% by only adding 0.1 wt% MWNTs to the Mg alloy [12]. By using pure titanium powder coated with un-bundled multi-wall carbon nanotubes (MWCNTs) via wet process, powder metallurgy (P/M) titanium matrix composite (TMC) reinforced with the CNTs was prepared by spark plasma sintering (SPS) and subsequently hot extrusion process. The microstructure and mechanical properties of P/M pure titanium and reinforced with CNTs were evaluated. The mechanical properties of TMC were significantly improved by the additive of CNTs [13]. The strength of the rolled strips is evaluated for various wt% CNT samples. The Al–0.5 wt% composite strips exhibited enhanced mechanical properties [14]. Nanocomposite with 0.75 wt.% of CNT exhibit well dispersed and embedded nanotubes and the highest hardness and tensile strength. The observed 200% increase in the tensile strength attested the strengthening effect of the CNT and the efficiency of the new dispersion treatment (R3 route) [15]. The presence of grain size refinement and improved dispersion of the Ni–CNT reinforcements in the Mg matrix were also observed. These result in simultaneous enhancements of the micro-hardness, ultimate tensile strength and 0.2% yield strength by 41%, 39% and 64% respectively for the Mg/Ni–CNT composites in comparison with that of the monolithic Mg [16]. Aluminium reinforced with CNT using powder metallurgy, are ball milled, compacted in a die made up of die steel, and then sintered. Obtained specimens are subjected to hardness, compression and chemical tests, compared with that of pure specimens [17]. An increase in strength is witnessed without compromising weight by reinforcing using CNT [18]. Using powder metallurgy Aluminium 6063 and CNT are reinforced, ball milled, compacted using D-2 steel, sintered at 350°C to 550°C at a rate of increase of 50°C per hour. There was an increase in compressive strength with no improvement in hardness of the composite on addition of CNT [19]. Reinforcement of metal matrix with CNT manually leads to appreciable increase in mechanical properties without compensation on its weight, by employing ball milling, compacting using cylinder die made of steel and then sintering. Various tests like tensile, compressive, hardness and chemical show that mechanical properties were improved significantly when compared to unreinforced iron [20].

Vijaya Ramnath et al [21,22] found mechanical properties of aluminium matrix composite in which stir casting techniques was used to fabricate the materials.

2. Materials Used

The materials used are Iron powder and carbon nanotubes. Iron powder is an aggregate of iron particles with sizes of approximately 20-200 μ m. It is treated as a powder using the particle size distribution, apparent density, etc. as indexes. Whereas, a Carbon Nanotube is a tube-shaped material, made of carbon, having a diameter measuring on the nanometre scale. A nanometre is one-billionth of a meter, or about one ten-thousandth of the thickness of a human hair.

3. Fabrication

1.1. Design of die

These standard practices cover the specifications for those uniaxial compacted test specimens that are used in ASTM standards, the procedures for producing and preparing these test specimens, and reference the applicable standards. Standard Practices for Production and Preparation of Powder Metallurgy (PM) Test Specimens. This standard is issued under the fixed designation ASTM B 925. PM test specimens are produced using the same methods as those used to make PM parts. Laboratory Tooling—If only a few test specimens are needed for the evaluation, they are usually produced using laboratory tooling. This may consist of a simple die supported on blocks and two plain punches or a laboratory tool set made with a spring loaded die and an adjustable lower punch. The compacting force is supplied by an ordinary hydraulic platen press or a compression testing machine. When compacting in laboratory tooling, filling, compacting and ejection are all controlled manually. Powder metallurgy tooling shall be made from materials that will resist the abrasive action resulting from compacting metal powder and ejecting green parts, but still have the mechanical strength to withstand the hoop stresses resulting from high compacting pressures. The tooling shall be precisely made and be capable of producing multiple identical test specimens. Before being used, all components of the tooling shall be free of grease or oil and be fully demagnetized. This annex describes the requirements of the components of the tooling used to compact PM test specimens. The fabricated die and punch is shown in Figure 1 and 2. This compression die is used to make a cylindrical specimen of height maximum of 20 mm. beyond this the densification of material getting low and also the tool may get damaged. The standard specimen as per the ASTM standard for different testing of P/M parts is of diameter 2.54 cm and 7.4 mm height. By using this we can get near net shape of the specimen.

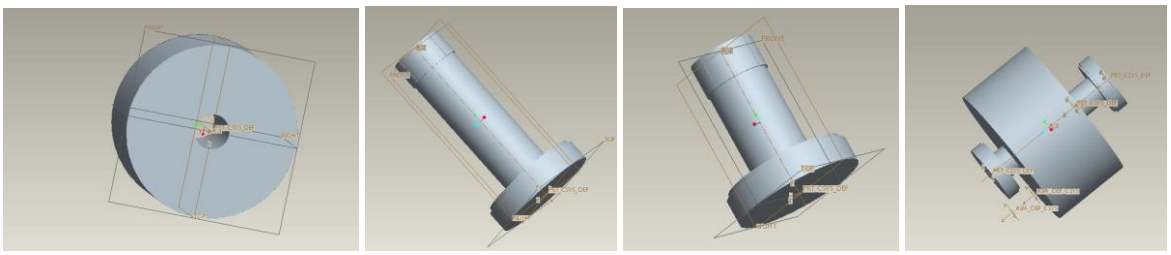

Fig. 1. 3-D view of compression die assembly

Fig. 2. Fabricated Compression Die

3.2. Compaction

Compaction of premix was carried out at different pressures from 400 Mpa to 600Mpa by using Hydraulic compacting press of capacity 100 tons. L/D ratio for samples was maintained less than 1 (L/D<1) accordingly dies and mass was selected. Die must me fixed and does not escape from the base plate when applying load. Initially the lower punch was inserted in the die case and placed properly in the base plate of hydraulic press. The blended material was measured as shown in the mass calculation and powder for different sample was kept separately. Zinc stearate was applied over the die cavity, upper punch and lower punch. Powder was poured into the die cavity carefully as the powder does not split over the surface. Load must be applied after the contact has been made between the upper punch

and upper plate of hydraulic press. The load has been applied gradually until required pressure has attained. After that the upper plate is moved upside and carefully the upper punch is removed. Compacted green samples were handled delicately and shifted to furnace for sintering operation. Unidirectional compaction was carried out at room temperature. Compaction force: 80 tons, Before Compaction (loose powder) = 15 mm. Compaction Process and Green specimen is shown in fig. 3(a) and (b) respectively. Pre Compaction (Zero load) =10.3 mm, After compaction (Full load) =9 mm.

(a) (b)

Fig. 3. (a) During compaction of compression specimen; (b) Compression after compaction

3.3. Sintering process

Sintering furnace of rectangular cross section (max Temp. 1250^{o} C) with a provision for necessary reducing atmosphere works as a Sintering furnace. Samples were placed in heating zone and heated to its sintering temperature (Ts= 0.8 - 0.9 Melting Point). During 1st step which is an intermediate heating step, the lubricant is removed and burned. This step is popularly known as 'de lube' or 'dewax' step. 2nd step is high heat step during which the powder particles fuse together, carbon from the premixed graphite dissolved in the ions diffuse into solid iron matrix with or without actually melting. The 3rd step of the sintering cycle is the cooling step. Both sintering and cooling are done in controlled atmosphere. Endothermic or N2 / H2 atmospheres are commonly used for sintering of steel parts. A typical sintering cycle for ferrous powder consists of preheating or DE waxing at 450° C (1202° F), sintering at 1120° C for 25 minutes in reducing atmosphere followed by cooling in the same atmosphere. The sintered specimen was removed from furnace. The same procedure was carried out for different composition of Iron- CNT. Sintered density: 6.6 g/cc

4. Testing of Composites

4.1. Compression test

A compression test is a method for determining the behaviour of materials under a compressive load. Compression tests are conducted by loading the test specimen between two plates, and then applying a force to the specimen by moving the crossheads together. During the test, the specimen is compressed, and deformation versus the applied load is recorded. The compression test is used to determine elastic limit, proportional limit, yield point, yield strength, and (for some materials) compressive strength. Testing can be performed on machined material samples or on full-size or scale models of actual components. These tests are typically performed using a universal mechanical testing instrument.

4.2. Micro hardness test

Micro Vickers hardness tester requires a micro-sized indenter, which allows hardness measurement in very limited areas such as surfaces of fine wires, thin sheets and foils. Moreover, hardness measurements at specific microstructural phases of materials, for instance, hardness measurement of ferrites and pearlites existing in steels is also possible. This is beneficial for identifying any hardness variation caused by metallurgical changes such as hardening, quenching, plating, welding, bonding processes, where the larger indenter used for macro Vickers hardness test limits its application in this case.

4.3. SEM

Scanning electron microscopy (SEM) is a method for high-resolution imaging of surfaces. The SEM uses electrons for imaging, much as a light microscope uses visible light. The advantages of SEM over light microscopy include much higher magnification (>100,000X) and greater depth of field up to 100 times that of light microscopy. Qualitative and quantitative chemical analysis information is also obtained using an energy dispersive x-ray spectrometer (EDS) with the SEM.

4.4. Metallography test

Samples for microstructure evaluation are typically encapsulated in a plastic mount for handling during sample preparation. Large samples or samples for macrostructure evaluation can be prepared without mounting. Sample preparation consists of grinding and then polishing using successively finer abrasives to obtain the desired surface finish. Sampling for metallography can be a random section to evaluate representative bulk properties or a section in a specific location to characterize localized material conditions.

4.5. Wear Test

The pin-on–disc wear testing machine was used for calculating wear rate of the samples. During testing the pin is kept stationary while the circular disc (also known as counter face) was rotated which cause friction between sample and counter face. Alumina disc (165mm diameter) was intentionally uses counter face. The surface roughness of the counter face was maintained by polishing it with 180 grit paper after every test. Acetone was used regularly before and after test to clean and remove traces of the disc.

5. **Results and Discussion**

5.1. Compression test

From the compression test result it is clearly revealed that increase in carbon nano tube, the compressive strength of the material also increased. 0.75 wt. % CNT yield a maximum compressive strength of 745 MPa.

5.2. Micro Vickers Hardness test

Micro Vickers hardness test was performed to determine the hardness of the specimen. Polished and machined specimen was tested for different trials and finally mean value is calculated. For 0.75 wt.% CNT have the high hardness value. Conversion of micro indentation hardness values to a familiar Rockwell scale may be done using Method B933. By using the conversion chart the Rockwell hardness value for the different specimen is shown in table 1.

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Table 1. Rockwell Hardness Value from Standard Table

Sample	HV	HRC
Pure Fe	328.66	33
0.25wt. % CNT	345.33	35
0.5 wt. % CNT	371.33	38
0.75wt. % CNT	423	43

5.3. Result of Wear test

The wear test results are shown in the table 2. It is observed that the abrasion loss is low when compared to pure iron samples. With 1 kg of load the wear rate is low and the same was high with 2 Kg of load. For both load test, wear rate is reduced with increase in carbon nanotube particles.

Wear test parameters

Cylinder size : 150mm Dia. & 500 mm Length

Material of coarser abrasive sheet : 60 Grade

Equivalent revolution : 84 times

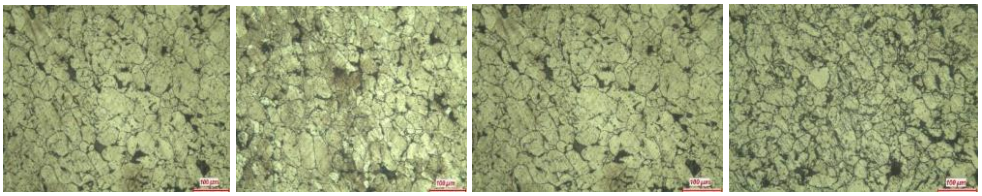
Rotational frequency : 40 $\pm$ 1 rpm

Table 2. Wear test result for different wt. % CNT

Sample	Load	Initial weight(*gm*)	Final weight (*gm*)	Abrasion Loss (*gm*)	In *%*
Iron	1 Kg	9.3923	9.0617	0.3306	3.52
	2 kg	9.0619	8.6197	0.4422	4.88
0.25 wt. %	1 Kg	9.4125	9.0859	0.3266	3.47
CNT	1 Kg	9.0869	8.6671	0.4198	4.62
0.50wt. %	2 kg	9.352	9.0331	0.3189	3.41
CNT	1 Kg	9.0416	8.6519	0.3897	4.31
0.75wt. %	2 kg	9.3712	9.0601	0.3111	3.32
CNT	1 Kg	9.0864	8.7248	0.3616	3.98

5.4. Microstructure

The microstructure of the various samples was performed in optical microscope to find the internal structure shown in fig. 4. (a), (b), (c) & (d). These samples were captured at 100 µm.

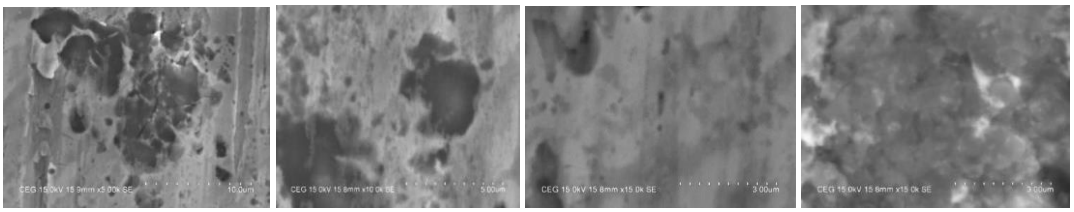


(a) (b) (c) (d)

Fig. 4. (a) Microstructure of pure iron at 100µm; (b) 0.25wt. % CNT at 100µm; (c)Microstructure of 0.50wt. % CNT at 100µm; (d) 0.75wt. % CNT at 100µm.

5.5. Morphological Analysis

SEM was performed to analyse the surface characterization of different iron and CNT samples shown in figure. fig.5(a) shows the pure iron specimen, fig. 5(b) shows 0.25 wt. % CNT, fig.5(c) shows 0.50wt. % CNT and fig. 5(d) shows 0.75 wt. % CNT. It is observed that micro crack was present in the sample and also voids present.

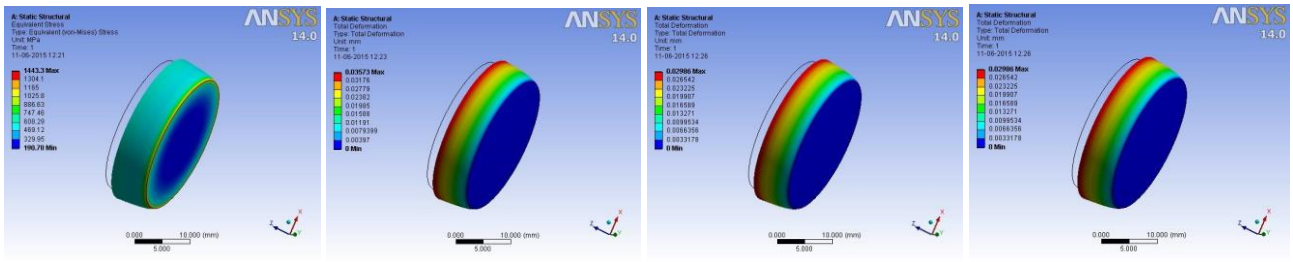


(a) (b) (c) (d)

Fig. 5. (a) Pure Iron (b) 0.25 wt. %CNT; (c) 0.50 wt. %CNT; (d) 0.75 wt. % CNT.

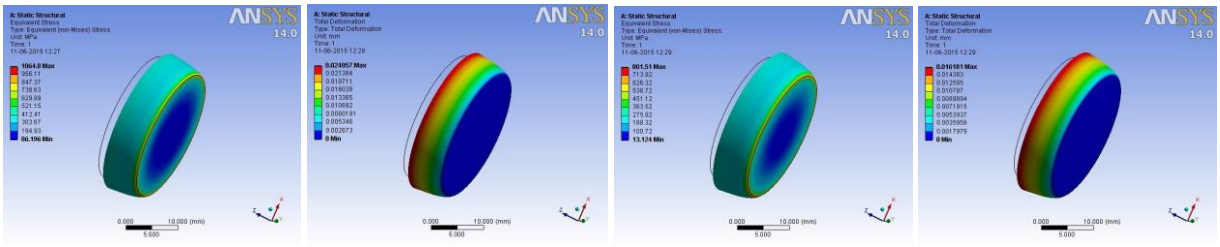
5.6. FEA

The FEA is done to predict the various stress and strain acting on the body. Analysis was carried out by ANSYS software. The material properties were assigned such as ferrous for pure iron, 0.25wt% CNT, 0.50 wt.% CNT and 0.75 CNT. The von- mises stress, total deformation as shown in fig. 6. (a), (b), (c), (d) and fig. 7. (a), (b), (c), (d).



(a) (b) (c) (d)

Fig. 6. (a) Pure iron- von-mises stress; (b) Pure iron- total deformation; (c) 0.25 wt.% CNT - von-mises stress; (d) 0.25 wt.% CNT – Total Deformation



(a) (b) (c) (d)

Fig. 7. (a) 0.50 wt.% CNT - von-mises stress; (b) 0.50 wt.% CNT – Total Deformation; (c) 0.75 wt.% CNT - von-mises stress; (d) 0.75 wt.% CNT – Total Deformation.

From the Finite Element Analysis, for both von-mises stress and total deformation, reinforcement of CNT will reduce the stress acting on the specimen and also the deformation due to the load will also less when compared to the pure iron. So, by comparing the von-mises stress for different CNT proportion, 0.75 wt. % CNT have the low stress and also deformation.

6. CONCLUSION

From the experimental results, increase in compaction load will increase the density of specimen and material properties. Improper mixing and binder composition will lead to the poor compaction and breaking of specimen during compaction. Proper handling of green specimen gives the good finishing and reducing the secondary operation. Sintering atmosphere have the high influence on mechanical properties. The pure iron samples were fabricated and CNT of different composition were added to increase its properties. The compression test shows that the 0.75wt. % of CNT have the high compressive strength. The hardness value and the compressive strength is increased with increase in wt. % of CNT and have the highest at 0.75wt. % CNT. From the wear test for iron and Nano iron specimen, for 1 kg of load, increase in wt. % CNT slightly reduces the wear rate and for 2kg of load increase in CNT will rapidly reduce the wear rate when compared with the pure iron. FEA results show that the deformation and von - mises stress is low for 0.75wt. % CNT. Thus iron with 0.75 wt. % CNT is selected as good combination with better mechanical properties by powder metallurgy method.

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