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Experimental & Numerical Investigations for Carbon Nanotubes Reinforced in Cement-based Matrix Composite Beams using FEM

Shankar A. Hallad[1], N. R. Banapurmath[2], Anand M. Hunashyal[3], N. H. Ayachit[4], Tajammul Husain[5], A.S.Shettar[6]
Centre for Material Science[1,2,3]
Department of Mechanical Engineering[2]
Department of Civil Engineering[3, 6]
B.V. Bhoomaraddi college of Engineering and Technology Hubli, Karnataka, India
Ranichannamma University Belgaum[4]
nr_banapurmath@rediffmail.com

Abstract:
The present work involves experimental and numerical investigations on the behavior of cement beams reinforced with multi-walled carbon nanotubes (MWCNTs) for structural applications. Specimens of size 20mmx20mmx80mm were prepared as per the ASTM standard with addition of MWCNTs in cement based matrix and the percentage of MWCNTs were varied with 0.25, 0.5, 0.65, 0.75, 0.85 and 1 per cent by weight of cement. Dispersion of MWCNTs in cement based matrix was carried out using ultrasonic energy. Composite beams were then tested under flexure in order to evaluate their mechanical properties such as flexural strength and load–deflection criteria. Numerical investigations using 3D finite element analysis for cement beams reinforced with MWCNTs were carried out. The results of experimental and numerical investigation were then compared with those from plain cement control beams. The present work also investigates the optimum percentage of MWCNTs that gives the best results in terms of both enhanced properties and economy. Scanning electron microscopy was conducted to examine the bond between the MWCNTs and the cement matrix. The numerical results were in good agreement with experimental results with an error of less than 9%. From analysis it has been observed that an optimum percentage of 0.75 has yielded the best performance results in both the investigations.

Key Word and Phrases
Multi Walled Carbon Nanotubes, Cement Composites, Three Point Loading, Finite Element Method

1. Introduction

Failure in many construction materials at the micro scale is typically the result of tiny micro cracks. Plain concrete is a brittle material and although it is not normally designed to resist direct tension, knowledge of the tensile strength of concrete is of value in estimating the load under which cracking will develop [1]. Advanced technological aspects have increased the demand for higher-performing cement-based materials with improved tensile strength and toughness for use in civil engineering projects. Cementitious materials are typically characterized as quasi-brittle materials with low tensile strength and low strain capacity and hence their use may affect the long-term durability of structures. These days, discrete short fibers are widely used to control cracking in fiber-reinforced concrete [2]. Nanotechnology is one of the most active research area, with both novel science and useful applications that have gradually established its importance over the past two decades [3, 4]. It enables scientists to work at the molecular level atom by atom to develop new materials with fundamentally new physical and chemical properties [5]. Carbon nanotubes (CNTs) are the subject of one of the most important areas of research [6]. CNTs were discovered in 1991 by Iijima in Japan [7] and were found to possess a Young's modulus of 1 TPa, a yield stress of 100– 300GPa [2] and a tensile strength of 63GPa [6, 8]. Nanoscience has a central role to play in producing innovative concrete materials for the 21st century. The incorporation of MWCNTs in cement increases the stiffness of the calcium silicate hydroxide (C-S-H) gel, resulting in a stronger material [9]. While the exploration for various applications of nanotechnology to develop

innovative construction materials continues, it is already clear that the science of the very small is making big changes, with numerous economic benefits for the construction industry [10]. By using CNTs, a new material which exhibits enhanced tensile strength and Young's modulus and improved early-age strain capacity can be created [11]. The various contributors to the research work on the utilization of CNTs in cement-based matrices are presented in Table 1 [12–16].

Researchers	Flexural strength increment (%)
Li et al. [12]	25
Cwirzen et al. [13]	20
Konsta-Gdoutos et al. [14]	25
Konsta-Gdoutos et al. [15]	40
Hunashyal et al. [16]	43.75(0.75% MWCNTs)

Table 1 Increment in mechanical properties with incorporation of MWCNTs for cement-based material

Hydrated cement is porous with a pore size distribution that ranges from nanometres to millimetres. The dimensional range of solids and pores in a hydrated cement paste has been reported in the literature [1]. The pore size classification of concrete includes microspores of radius less than 1.25 nm, mesopores of radius 1.20–25 nm and macrospores of radius 30–5000 nm. The pores are an important component of the microstructure of a cement-based composite and have a great influence on the strength and durability properties of the material. A number of research groups have investigated the influence of CNTs on the pore structure of ordinary Portland cement (OPC) and CNT composites. The findings of the studies suggest that the combination of hydration product nucleation and pore bridging may result in a densification of the OPC/CNT composite and a reduction in the overall porosity and pore continuity of the material. However, two problems associated with the addition of CNTs and carbon fibers to any material include clumping together of the tubes/fibers and the lack of cohesion between them and the bulk matrix material. A uniform dispersion must be achieved to get the desired results [8]. The cost of adding CNTs to concrete may be prohibitive at the moment but work is being done to reduce their price and at such time the cost/benefit ratio offered by their addition to cementitious materials may become more acceptable and economical as well. Most of the published papers on the use of MWCNTs in a cement matrix have focussed on preliminary work involving very low additions varying from 0.08 to 0.25 per cent by weight of cement, keeping in mind the cost and dispersion issue of MWCNTs.

Studies using a higher percentage of (0.75% MWCNTs) MWCNTs are desirable but the difficulties encountered in the dispersion of MWCNTs hindering the research. Experimental work [2] has shown that addition of 0.25 wt% of MWCNTs to plain cement beams results in remarkable enhancement in their flexural strength and toughness properties. In comparison with plain cement beams, the CNT-reinforced beams showed a 47 per cent increase in load-carrying capacity and an average increase in toughness of 25 per cent. Metaxas et al. [17] tested CNT/cement composites under flexure and found that, with proper dispersion, the fracture properties of cement matrices can be substantially increased by adding a very low amount of CNTs (i.e. 0.025 to 0.10 per cent of MWCNTs by weight of cement). Scanning electron microscopy (SEM) has shown that the addition of a small amount of CNTs enables the control of matrix cracks at the nano scale and nano-indentation tests have suggested that CNTs can modify and reinforce the cement paste matrix at the nano scale. The foregoing results suggest that CNTs have potential to be used as reinforcement for concrete; however, there is still a significant need for further research studies in this area to completely understand the structural behavior of CNT-reinforced cement [18, 19]. Fundamentally, hydrated cement paste is a nanomaterial. Knowledge at the nano-scale level of the composition and characteristic of materials will promote the exploration of new applications and new products that can be used to repair or to improve the properties of construction materials [18]. It is reported that compression strength of the cement paste increases with the addition of CNTs that decreases the

number of mesopores [20]. CNTs have the potential to enhance the strength, effectively hinder crack propagation in cement composites, and act as nucleating agents [4]. Reinforcing concrete with CNTs will produce tougher concretes by interrupting crack formation as soon as it is initiated. The interactions of CNTs in a cement matrix that hinder crack propagation have been reported in the literature [5]. The present work utilized MWCNTs, having diameters of 10–30nm, densities of 1000–2000 kg/m3, and an aspect ratio of 1000:1, to produce reinforced cement beams. The addition of MWCNTs was varied from 0.25 to 1 per cent by weight of cement, and dispersion of MWCNTs was carried out using ultrasonic energy. The mechanical properties of the composite beams were tested and the results were compared with those from plain cement control beams. The optimum percentage of MWCNTs that gives the best results in terms of both enhanced properties and economy was investigated. SEM was conducted to examine the bond between the MWCNTs and the cement matrix.

Since laboratory testing of CNT/cement composite placed a number of challenges on thoroughly assessing the behavior of the composite, the further study focused on the application of finite element method (FEM) as an alternative to the experimental investigation. There has been a debate among scientists and researchers on whether continuum mechanics approaches could be utilized in studying materials on the nano level. Based on numerous studies [28–29], there has been an agreement that the theory of continuum mechanics could be utilized in situation where a global mechanical property is of interest such as determining Young's modulus or global deformations of CNTs or CNT-based composites. However, if the interest is in studying the local behavior of the material such as obtaining local stresses or strains in the vicinity of a single atom or even a group of atoms, then molecular dynamic (MD) simulations or experimental methods should be used. It should be noted that in the present study, authors were mainly interested in studying the global behavior of CNT/cement composites and in what way the introduction of deficiencies in the averaged bond strength between CNT and the surrounding cement matrix would affect the global behavior. Therefore, no attempts were made in this paper to investigate the local bond characteristics of CNT and cement on the atomic level. In addition, the study also addressed correlating the mechanical properties obtained from a nano-scale finite element (FE) model with the macro behavior of a structural element built with CNT/cement composites.

This paper utilizes finite element (FE) models to study the improvement in flexural strength of cement when using carbon nanotubes (CNTs) as reinforcement. Nano scale and macro result models are used to investigate the interfacial bond behavior of an individual CNT embedded in cement matrix and flexural response of a CNT/cement composite beam, respectively. Experimental test results of CNT/cement composite beams were used to compare the numerical models. The results prove that FE results are in close tolerance with the CNT/cement composite beam strength and its effects on flexural strength.

2. Experimental Program

The properties of the MWCNTs used in the present study are given in **TABLE 2.**

Diameter	10–30 nm
Length	1–2 μm
Purity	95%
Surface area	350 m^2/g
Bulk density	0.05–0.17 g/cm

Table 2 Properties of the MWCNTs used in the present work, supplied by Intelligent Private Limited

The MWCNTs were of industrial grade with a purity of greater than 95 percent. Uniform dispersion of MWCNTs against their agglomeration due to vander Waals' bonding is the first step in the processing of nanocomposites. Dispersion is a critical issue while mixing CNTs in either

water or organic solvents. Ethanol was used for pre-dispersion of MWCNTs in cement using a sonication method, as suggested by Makar and Beaudoin [6]. Care was taken to ensure evaporation of the ethanol and later water was added without taking into account the amount of ethanol used when calculating the water-to-cement (w/c) ratio of the paste. Moreover, the method of sonication, duration of sonication and method of casting the specimens were maintained uniformly throughout. Different amounts of MWCNTs were added to cement as shown in TABLE 3 and the whole mixture was kept in an ultrasonicator for 90min to achieve satisfactory dispersion [21].

Size	20mm×20mm×80mm
Water/cement ratio	0.4
Amount of CNTs	0.25, 0.5, 0.65, 0.75, 0.85, and 1 per cent by weight of cement

Table 3 Specimen characteristics

A w/c ratio of 0.4 was found to be appropriate in terms of mixing and workability of the paste after adding the CNTs. After this process, the paste was carefully placed into prepared moulds and compacted using a tamping rod. The cast specimens of dimensions shown in Table 3, were demoulded after 24 h, cured for 28 days and tested on 28th day. Serving as the control, plain cement beams without any reinforcement were also prepared. The designation of the test specimens used is shown in Table 4.

Sample no	Specimen Reference	Specimen details	
		Constituents	Percentage of MWCNTs by weight of cement
1	PC	Plain cement	Nil
2	A1	Plain cement + MWCNTs	0.25
3	A2	Plain cement + MWCNTs	0.50
4	A2*	Plain cement + MWCNTs	0.65
5	A3	Plain cement + MWCNTs	0.75
6	A3*	Plain cement + MWCNTs	0.85
7	A4	Plain cement + MWCNTs	1.00

Table 4 Details of the test specimens

Experimental tests were conducted to investigate the effect of the uniformly dispersed and randomly oriented MWCNTs in the cement paste as per the Bureau of Indian Standards [22–24]. For experimental accuracy, ASTM C 348 was followed to determine the average values of flexural strength (ASTM C 348 determines the flexural strength of hydraulic-cement mortars). Three-point bending tests as shown in Fig.1 were carried out.

Fig. 1 Placing of the specimen for testing

SEM was also conducted on all samples with varying percentages of MWCNTs used.

2.1 FE Modeling of Three Point Bending Test

A simply-supported-beam model was constructed in ANSYS [30] to investigate the flexural behavior of the CNT/cement composite beams tested in the laboratory to characterize CNTs reinforcing efficacy. The dimensions of the model were identical to the tested specimens (20 mm × 20 mm× 80 mm). Fig. 2 (a) and (b) shows the details of the FE model in ANSYS**.**

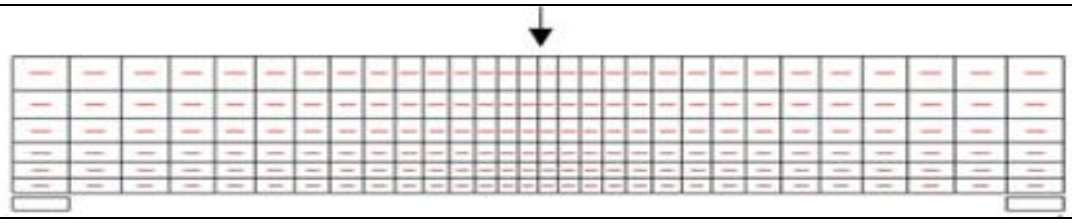

Fig. 2 (a) Side view

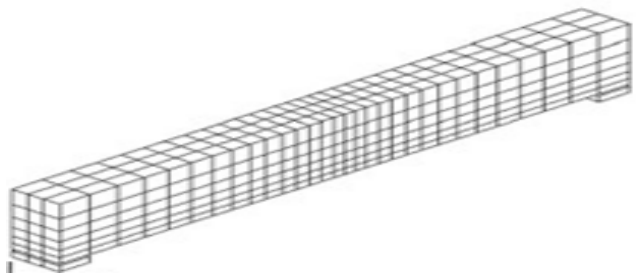

Fig. 2(b) Isometric

The beam was meshed using SOLID65 elements and was constructed to have the mesh refined toward both the mid-span and the bottom fiber, where cracking is expected to initiate. Smeared reinforcement, as illustrated by the broken lines in FIG. 2a of 0.25% by weight of cement was defined to be oriented in the longitudinal direction. However, the effect of CNT random orientation will be accounted for implicitly in the effective shear strength produced from calibrating the FE model with the experimental results. The material properties of cement were modified based on the experimental results of the plain cement beam to make a more legitimate comparison of the numerical results and the experimental response. The beam was loaded at mid-span under displacement-controlled setting. A number of trial simulations were run to determine the number of elements needed to balance the computational cost and the accuracy of the analysis. A total number of 3462 elements and 5064 nodes were employed in the final beam model. The primary goals of the study considered different numerical results corresponding to different proportions of the MWCNT in 0.25, 0.5, 0.65, 0.75 and 1 percent respectively. The smeared reinforcement in the simplified model was constructed by defining its volume ratio in the cement matrix and its material type under element properties of SOILD65.

3. Results and Discussion

The objective of the present study was to find the optimum percentage of CNT dosage and the effect of CNTs on the ultimate load-carrying capacity of the MWCNT-reinforced cement composite under three-point loading. Fig .3 shows the load–deflection curves for composites with different percentages of MWCNTs embedded in the cement matrix**.**

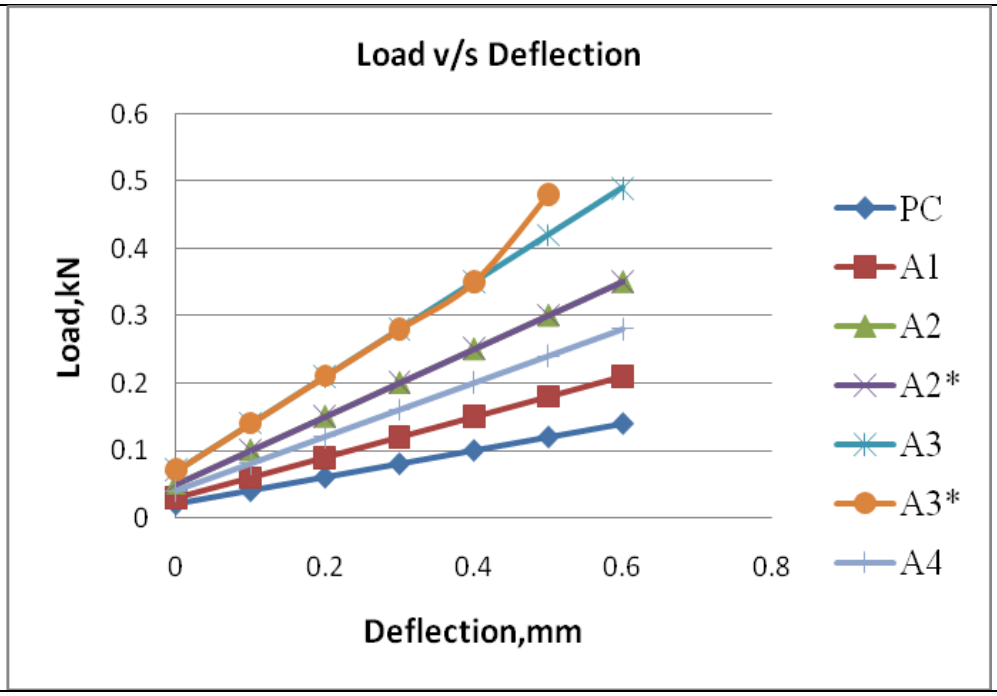

Fig. 3 Load–deflection curves for the specimens in three point loading

The ultimate load was calculated for composites containing 0, 0.25, 0.5, 0.65, 0.75, 0.85 and 1 per cent of MWCNTs by weight of cement as shown in Fig.4.

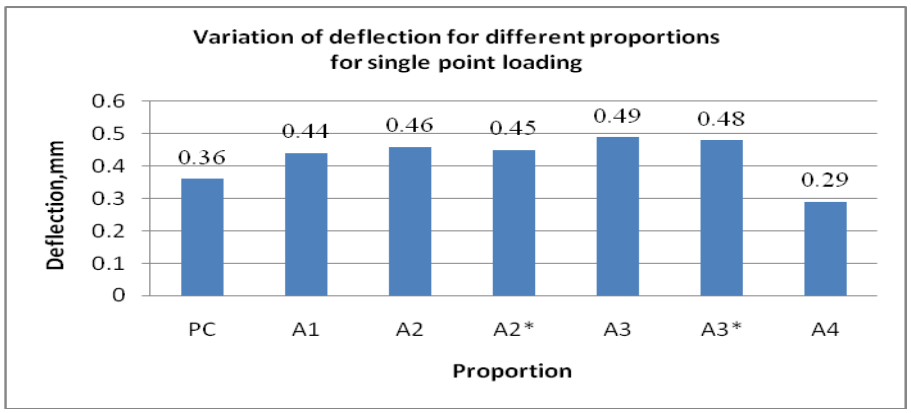

Fig. 4 Variation of deflection for different specimens

The variation of flexural strength was studied keeping PC beams as reference and the flexural strength followed an increasing trend up to 0.75 wt% of MWCNTs as shown in Table 5**.**

Sample no.	Specimen Reference	Ultimate load (kN)	Maximum deflection (mm)
1	PC	270	0.36
2	A1	280	0.44
3	A2	340	0.46
4	A2*	360	0.45
5	A3	500	0.49
6	A3*	490	0.48
7	A4	140	0.29

Table 5 Test results for the MWCNT-reinforced cement composites

Specimen A3 with 0.75 wt% MWCNTs showed a maximum ultimate load of 500 N, while the reference beam gave a value of 270 N. Since the composite, at greater MWCNT contents, has a tendency to undergo large deflections as shown in Fig.5 the CNTs provide additional toughness to the composite.

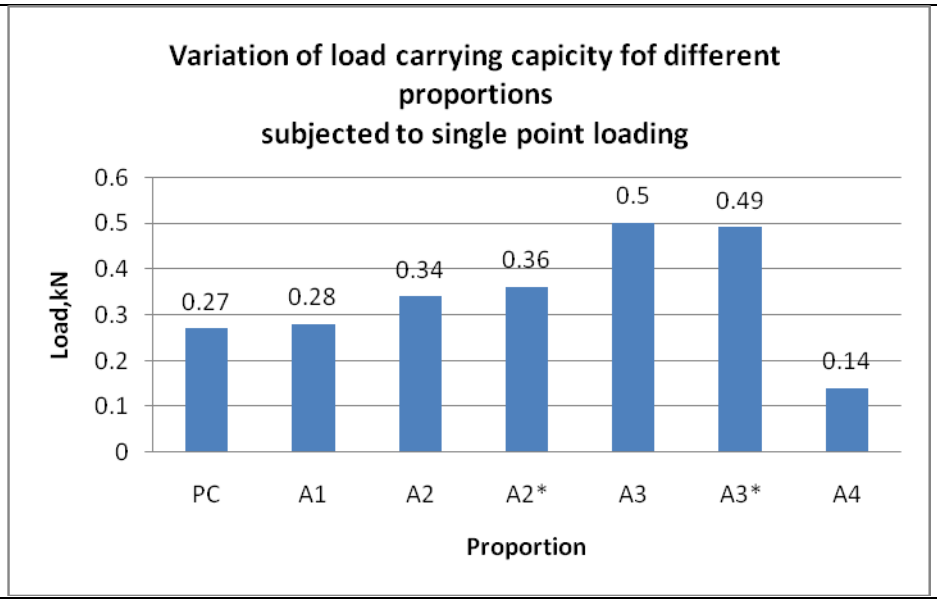

Fig. 5 Variation of the ultimate load of the specimens in single-point loading

The energy-absorbing capacity of the composite generally increased, i.e. a greater amount of load is carried since the CNTs resist the crack propagation. The mechanical properties of CNTs have drawn intense interest owing to their potential for use as reinforcements in composite materials. In addition to their strength and elastic constant, CNTs have extremely high aspect ratios, with values typically higher than 1000:1 and reaching as high as 2 500 000:1 [1]. The size and aspect ratio of CNTs mean that they can be distributed on a much finer scale than commonly used reinforcing fibers. As a result, cracks are interrupted much more quickly during propagation in a CNT-reinforced matrix, producing much lower crack widths at the point of first contact between the moving crack front and the reinforcement. As a result of these properties, CNT reinforcements are expected to produce significantly stronger and tougher composites than traditional reinforcing materials.

Fig. 6 shows a comparison between experimental and numerical values of deflection for CNT/ cement composite with 0.75 vol% CNTs, from which it follows that CNTs act as bridges across pores and cracks.

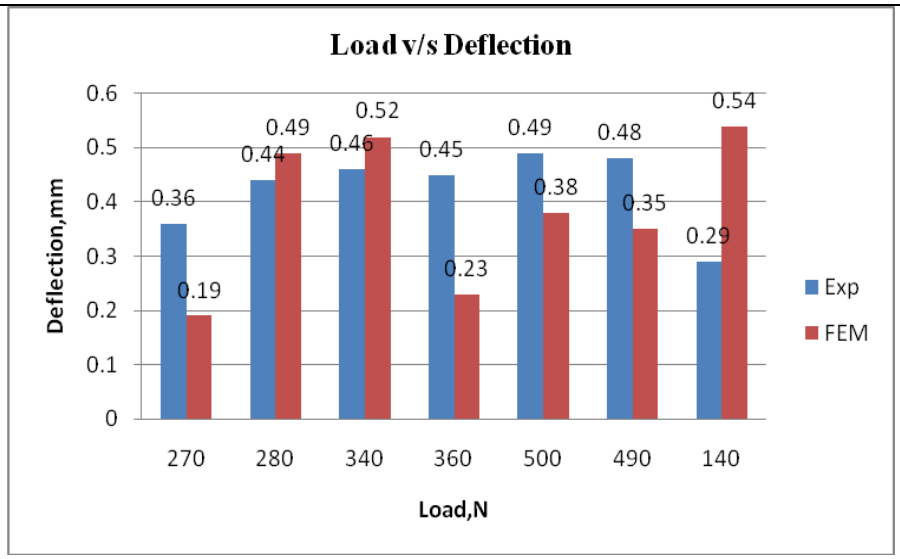

Fig. 6 Variation of Experimental and FEM Deformation for thee point bend specimen

FEM analysis is carried out using ANSYS. The model is created using brick element of solid65. Material property is calculated using rule of mixture :

$$\vartheta_1 = \vartheta_f V_f + \vartheta_m V_m \tag{3.1}$$

$$E_1 = E_f V_f + E_m V_m \tag{3.2}$$

For different proportion of MWCNTs Young's modulus was calculated and corresponding Poisson's ratio was arrived at. Before applying BC's the small strip area of width equal to the support of three point bend specimen were created. Motion of supported area is restricted parallel to the loading direction. Load is applied on the nodes at the mid of the specimen by selecting proper elemental area. Solution of the corresponding cement beam changed from linear to non linear behavior indicating plastic deformation occurred in specimen and deformation for various combinations of MWCNTs and cement matrix was then calculated, with approximate solution for every combination of ultimate load with an error less than $\pm 9\%$ was found.

The load v/s deformation characteristic of reinforced beam as per FEA analysis is shown from Fig. 7 to Fig. 13. From these figures it follows that maximum deformation is observed with A3 samples having higher percentage of MWCNTs compared to other samples considered. However for A3* sample decrement in deformation was observed and these results are in consistent with the experimental results obtained as well.

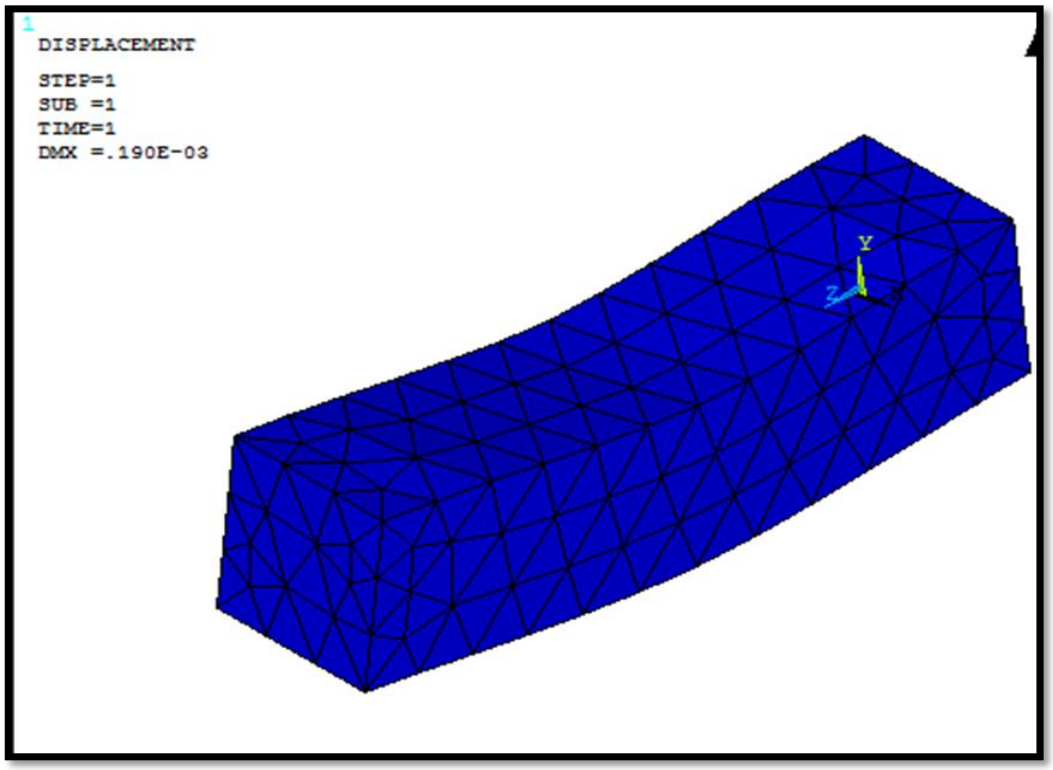

Fig.7 PC

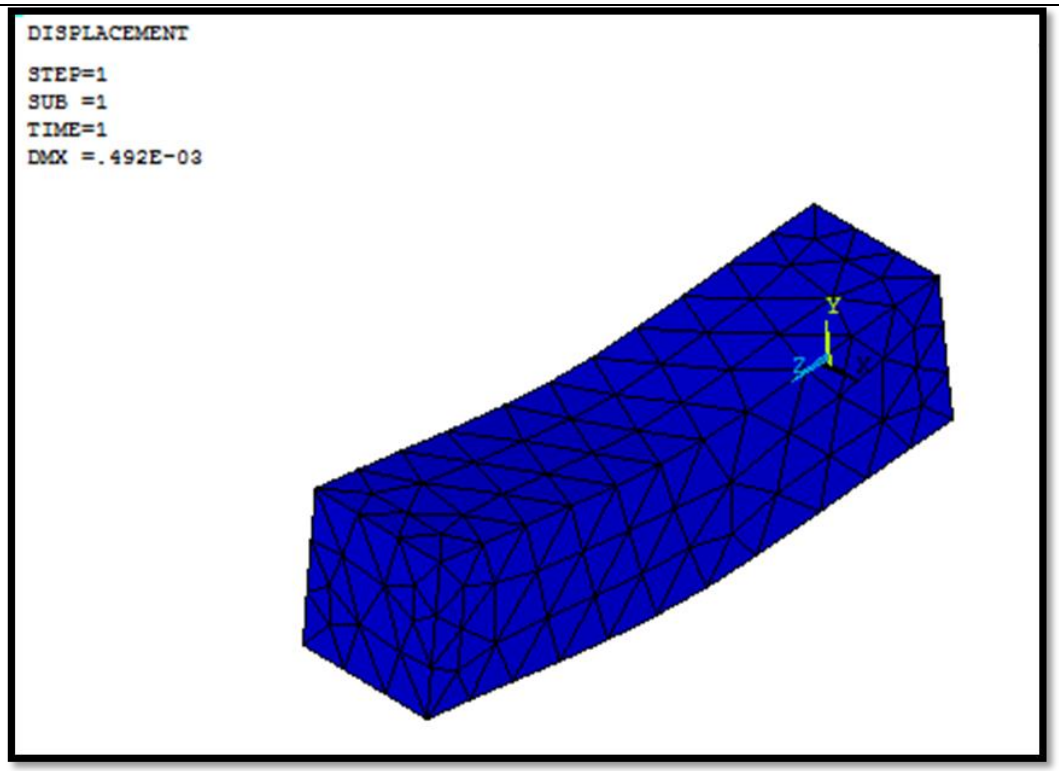

Fig. 8 A1

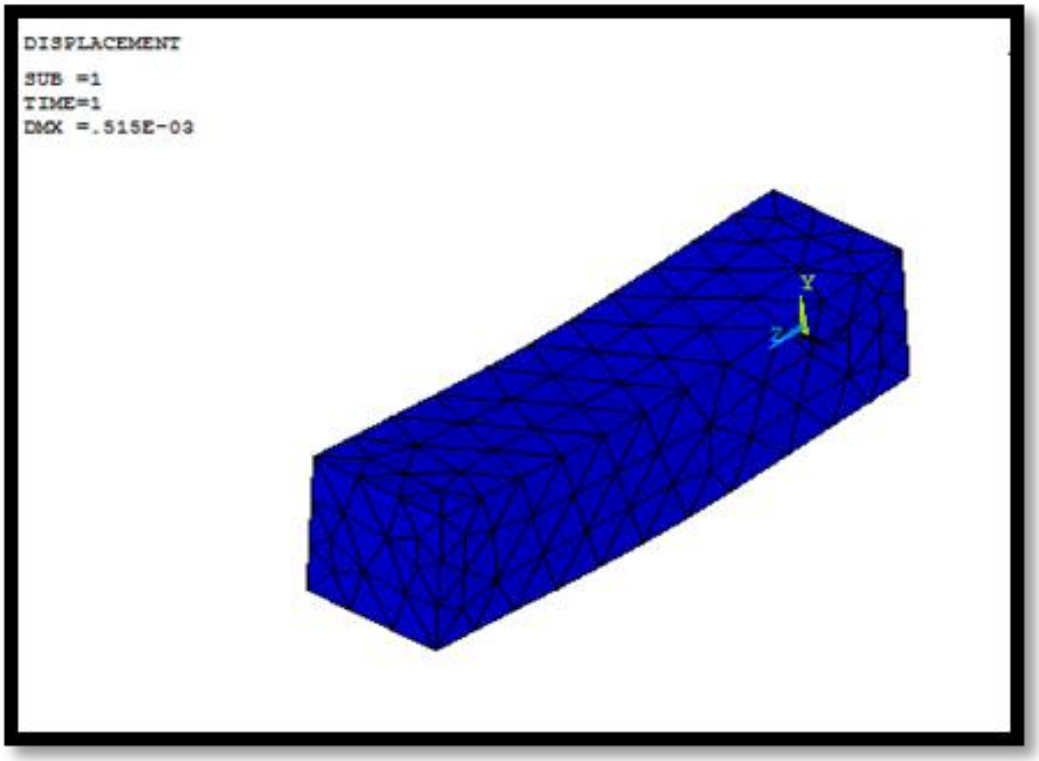

Fig. 9 A2

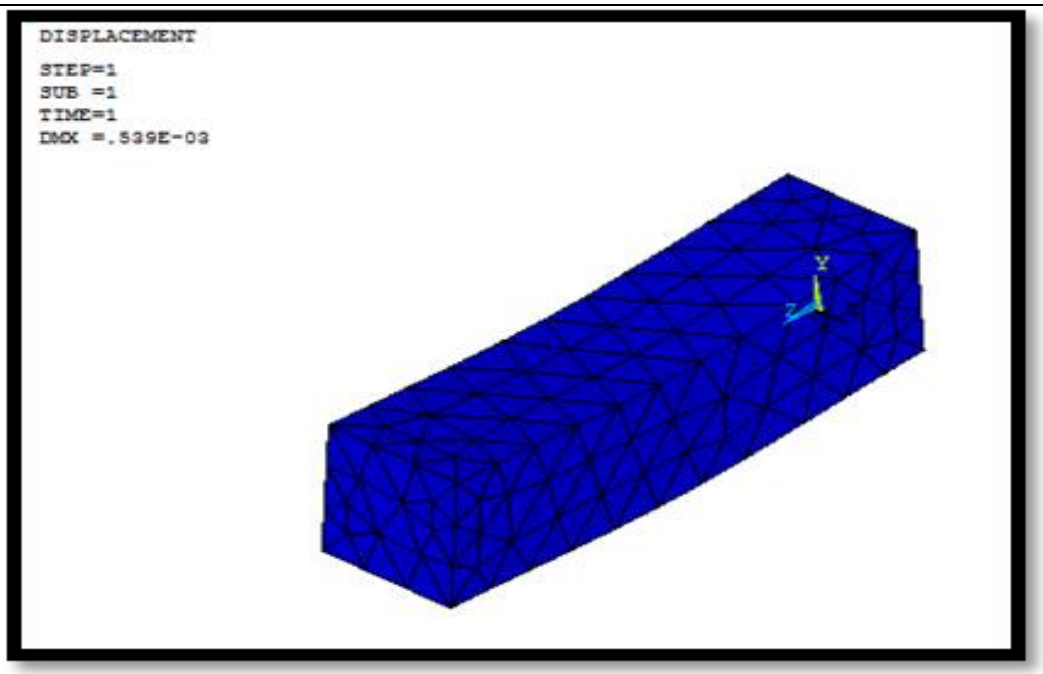

Fig.10 A2*

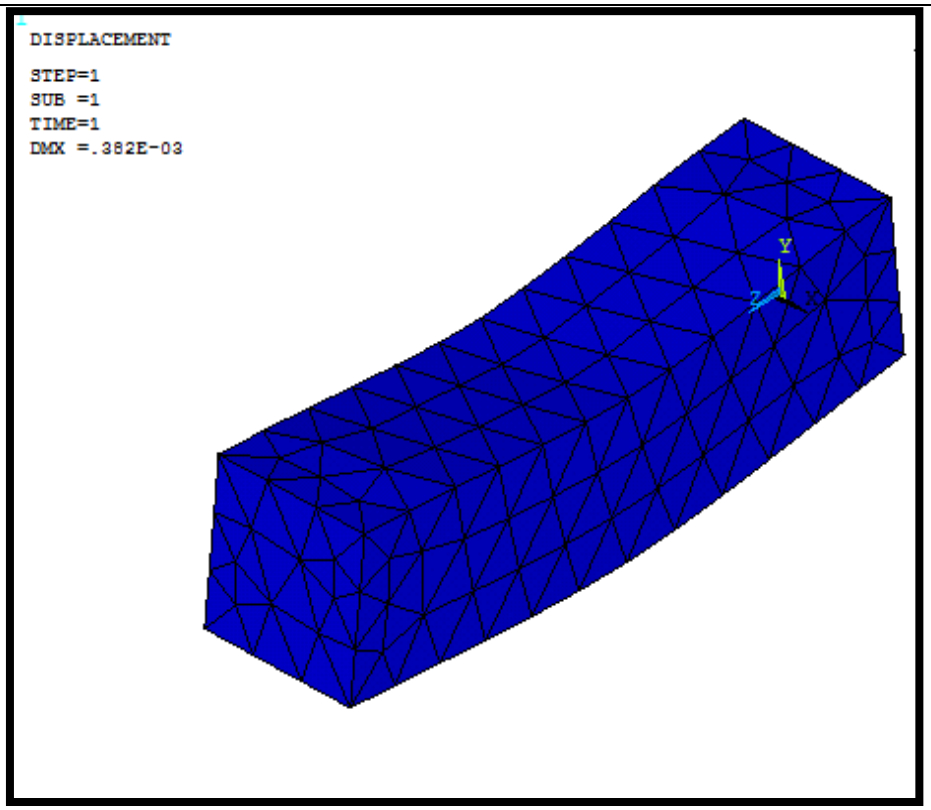

Fig. 11 A3

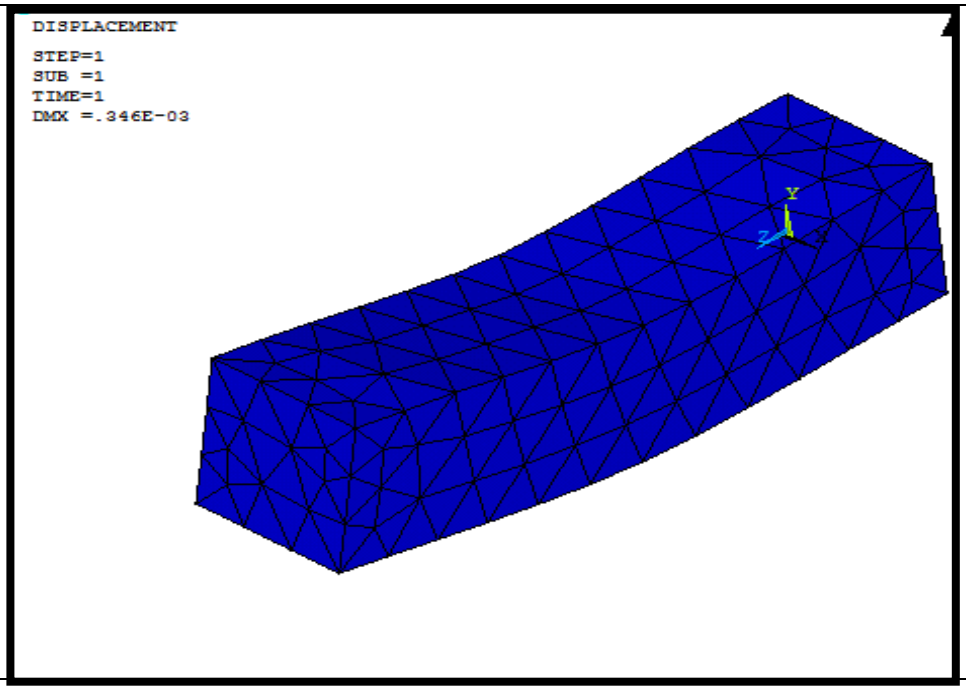

Fig. 12 A3*

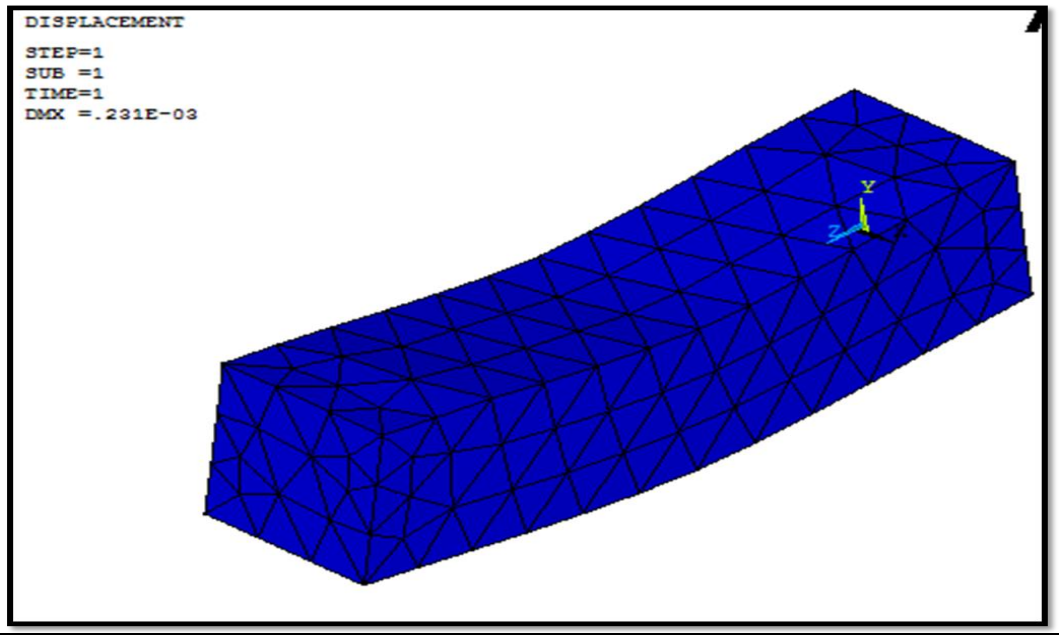

Fig. 13 A4

The Von-Mises stress criteria which follow the principle of continuum mechanics for different composition are shown in Fig. 14 to Fig. 20**.** From these figures Von-Mises stress criteria further confirms the justification for the above deformation observed in the respective samples tested.

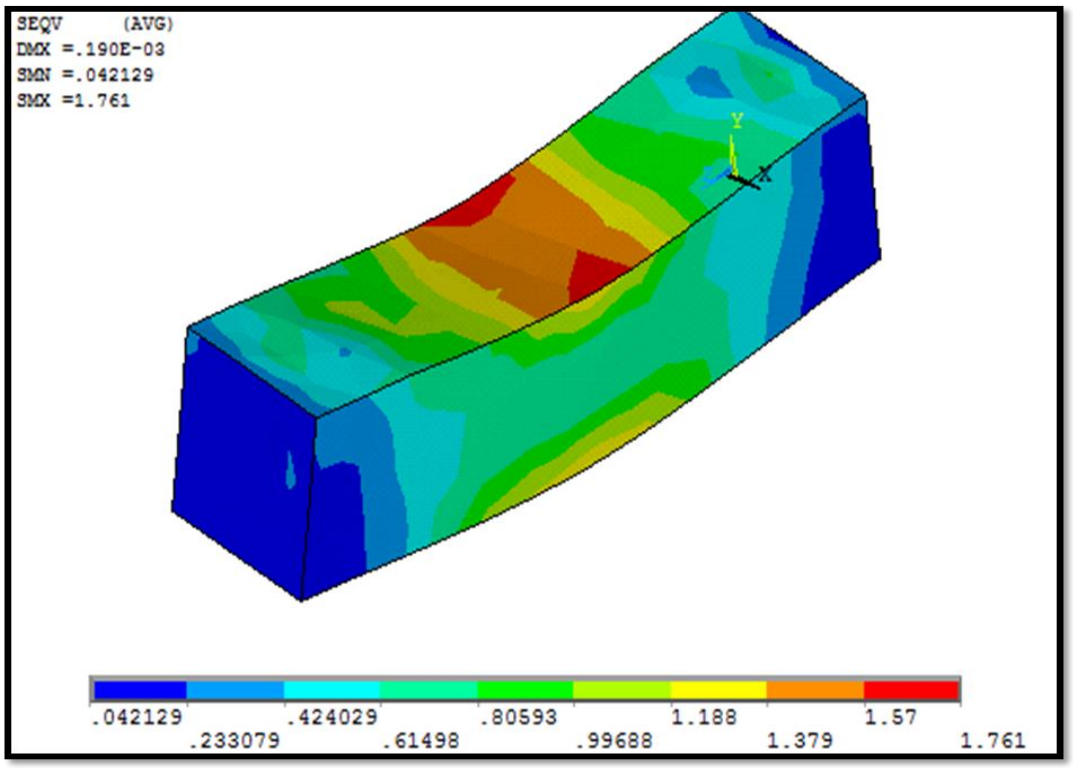

Fig. 14 PC

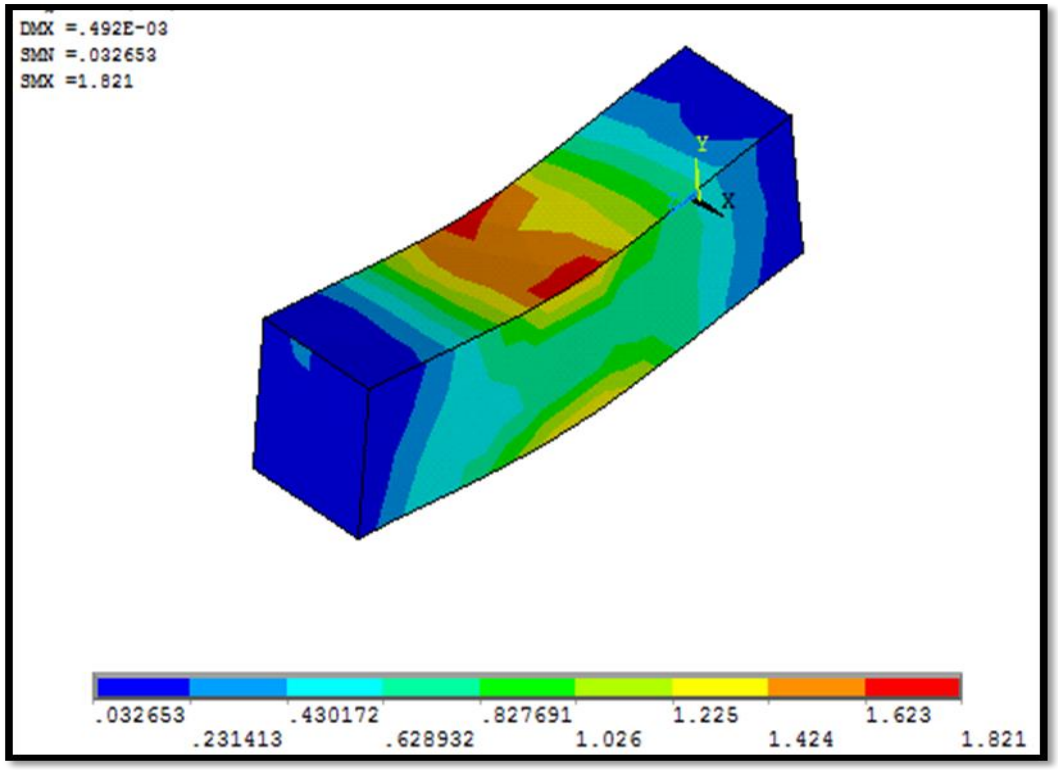

Fig. 15 A1

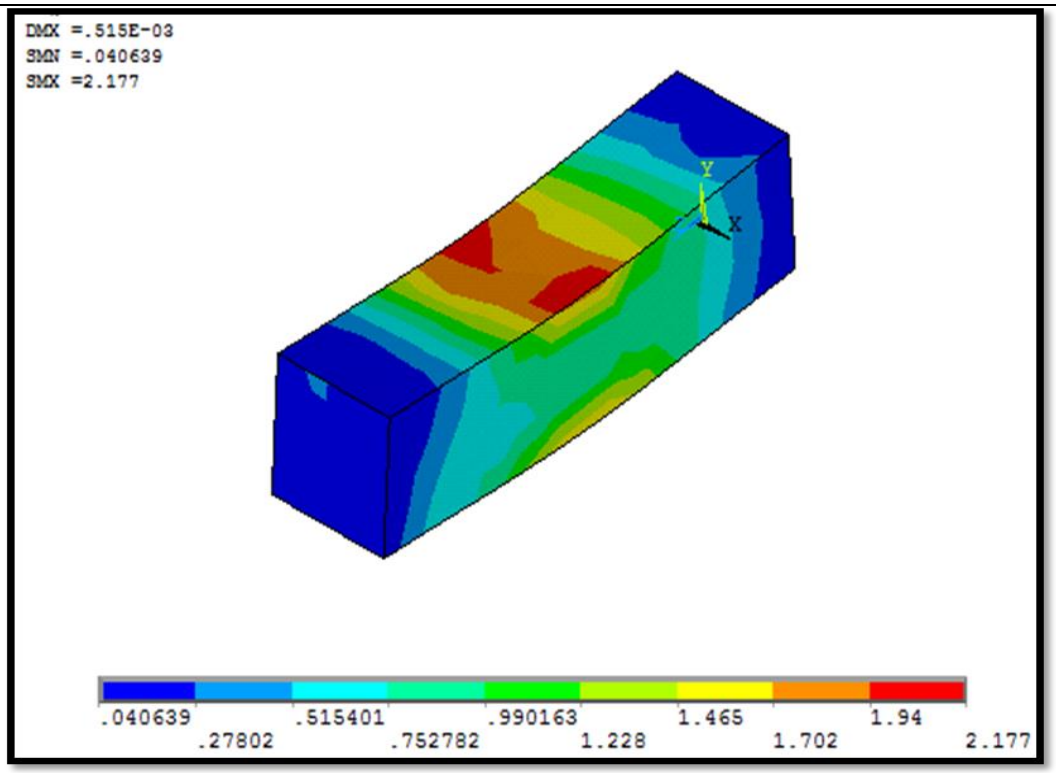

Fig. **16** A2

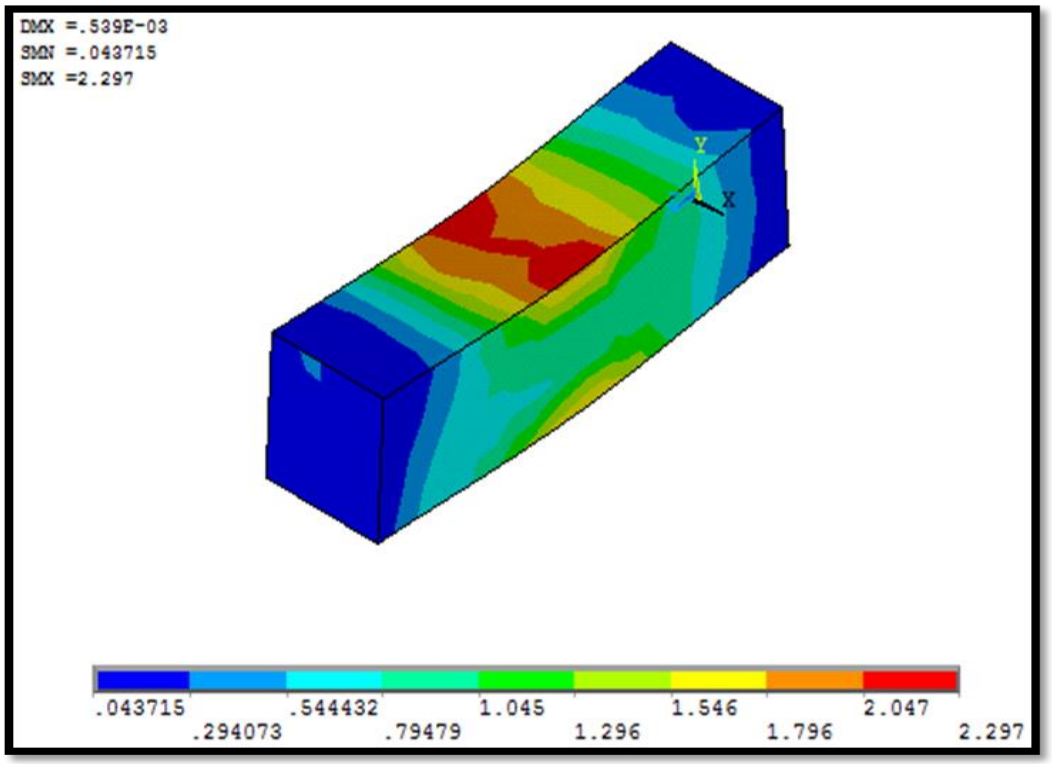

Fig. 17 A2*

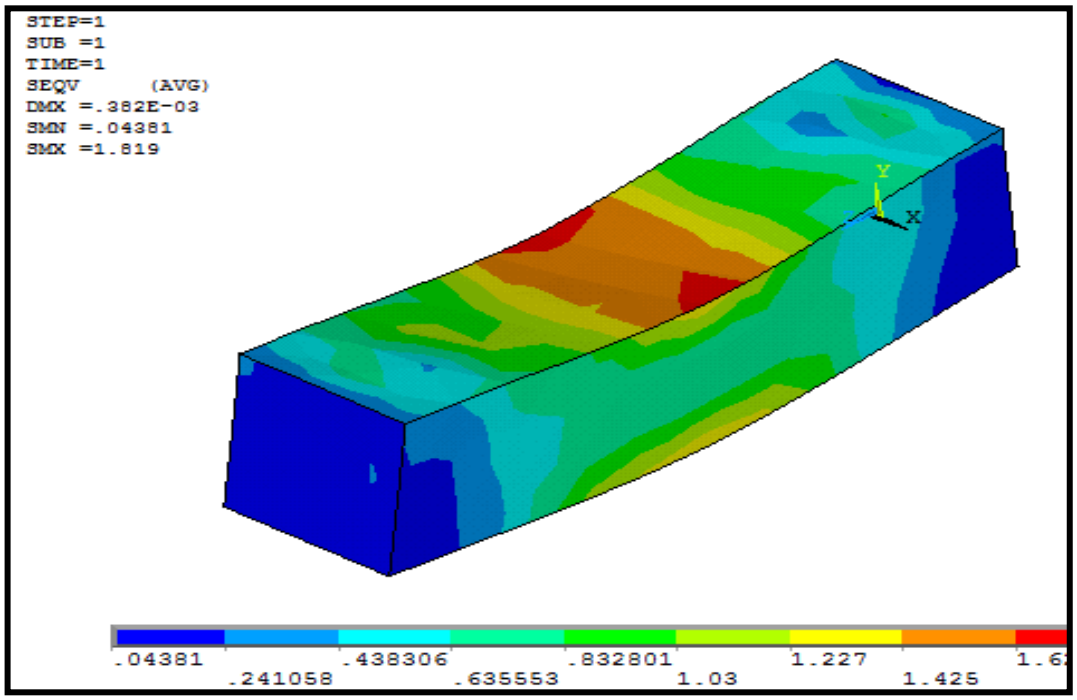

Fig. 18 A3

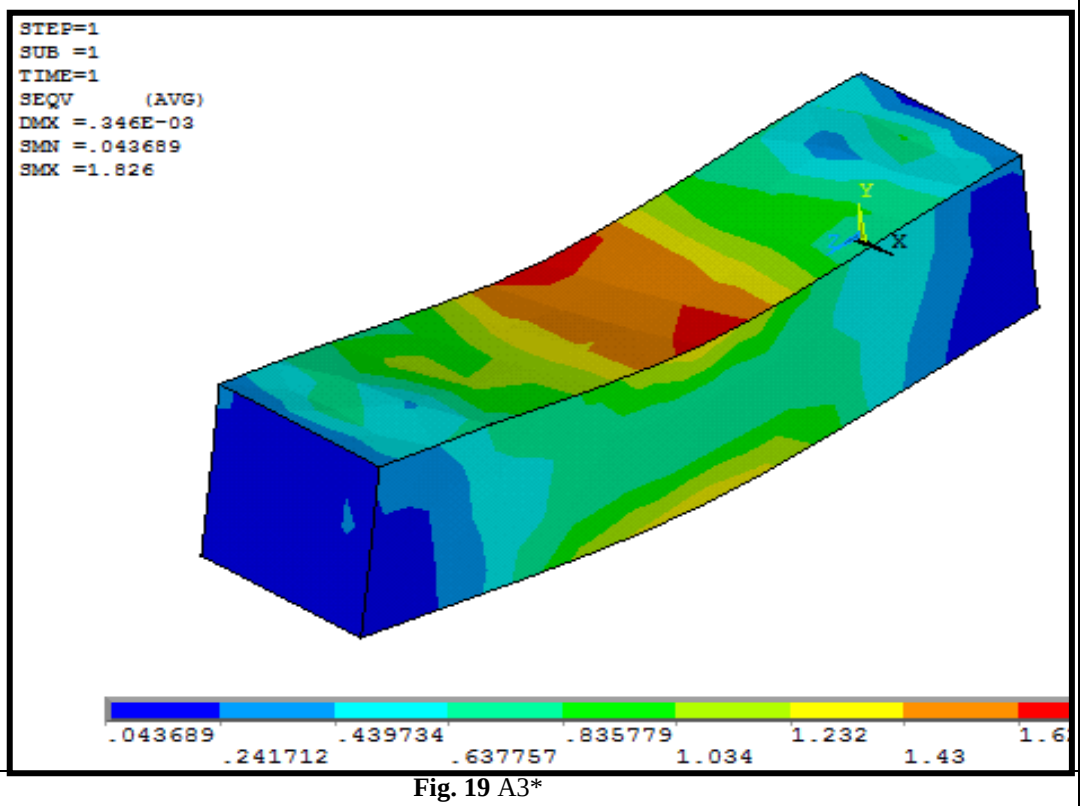

Fig. 19 A3*

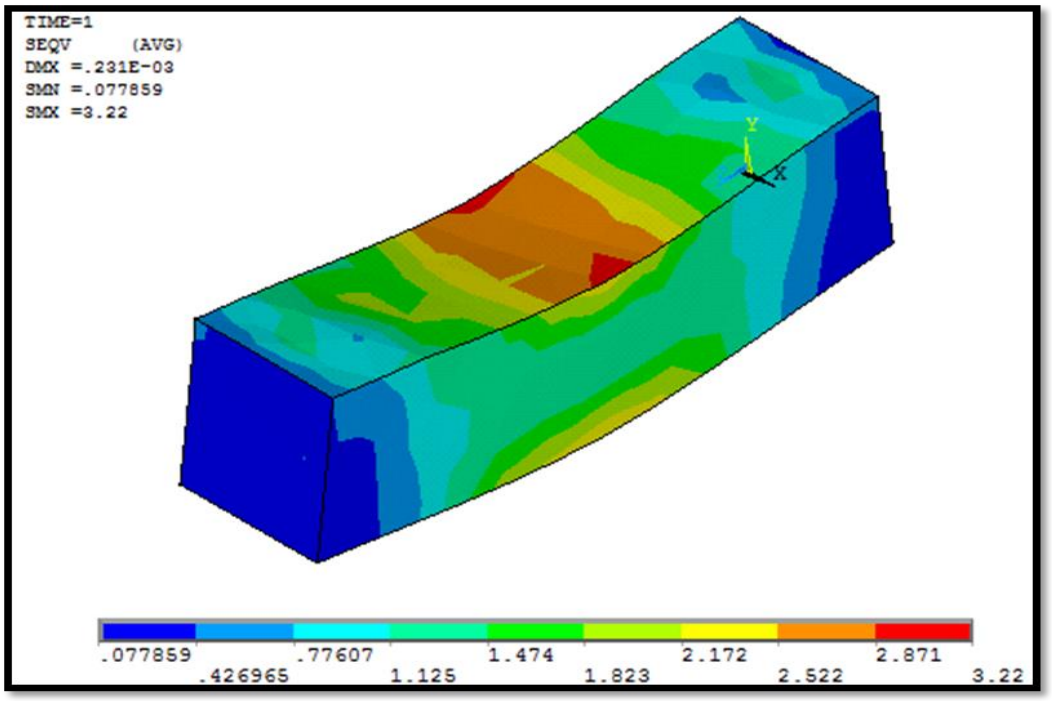

Fig. 20 A4

Fig. 21 shows a SEM micrograph of the CNT/cement composite with 0.75 wt % of CNTs which reveals interaction between cement matrix and MWCNTs. From the Figure it appears that the CNTs are tightly wrapped by C-S-H bonding. This indicates high bonding strength between the CNTs and cement matrix was achieved. This is consistent with other results published in the literature [25],[26].

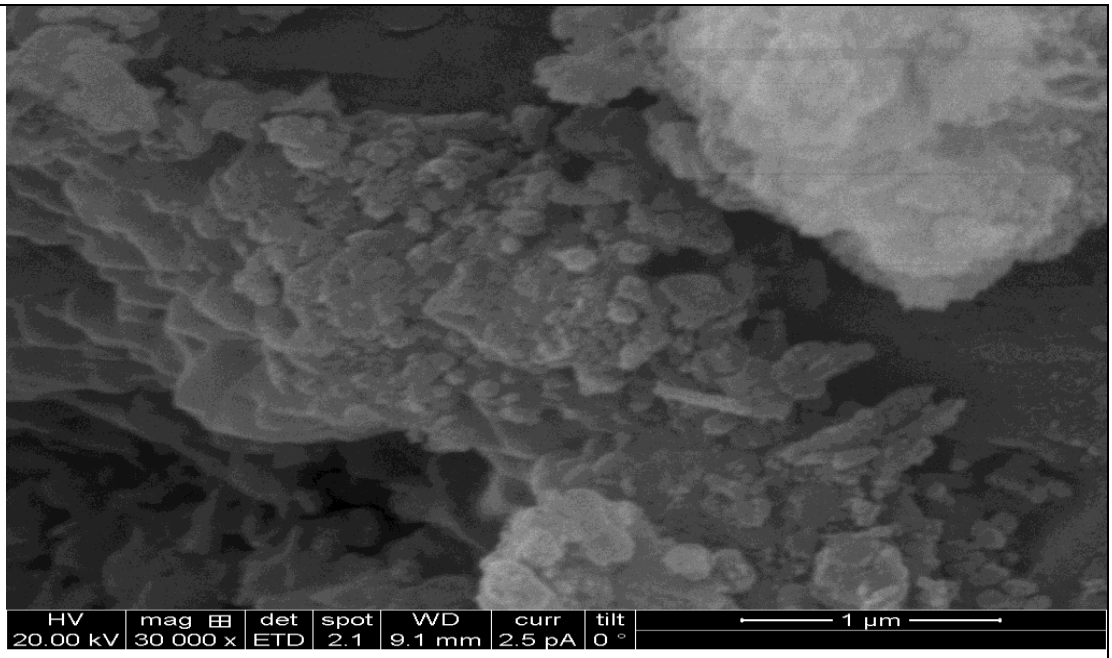

Fig. 21 SEM micrograph of the MWCNT/cement composite with 0.75 wt% CNTs

4. Conclusions

Experimental observations revealed that the MWCNT-reinforced cement composite beams showed increased strength compared with plain cement beams. The nano-level reinforcement significantly improved the flexural strength of the beams [26], [27]. The results showed an increase in the load carrying capacity of the composite beams compared with the reference beams.

1. Compared with the plain cement control beams the flexural strength of the cement composite was increased by 30.76, 37.93, and 43.75 % with MWCNT additions of 0.25 wt%, 0.5 wt%, and 0.75 wt%, respectively.
2. Composites with higher weight percentages of MWCNTs showed higher deflections. They underwent large deformation with increased load-carrying capacity. This may be due to inhibition of cracks by the MWCNTs. The composite with 0.25 wt% MWCNT content showed a lesser amount of deflection compared with the other nanocomposites.
3. Nanotube reinforcements will increase the toughness of composites by absorbing energy during their highly flexible elastic behavior. This proves their ability to become an ideal reinforcing material.
4. As the loading of MWCNTs increased, the distribution in the cement matrix was not uniform and this is unsatisfactory as it results in lower load carrying capacity. This happened particularly for 1wt% of MWCNTs added to the cement matrix.
5. As the % CNTs increased in cement matrix the strength further increased, this is observed till 0.75% by weight of CNTs and could be due to the high surface area of nanoparticles attracting the cement which reduces the mobility of C-S-H chains and hence causes increase of viscosity in the cement matrix.

6. For 1% CNTs by weight the strength decreased drastically and this could be due to increased nanotube waviness and may be an additional mechanism limiting the modules enhancement of nanotube-reinforced cement matrix. It has been observed that even slight nanotube significantly reduces the effective reinforced when compared to straight nanotubes.

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