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# CREEP AND HARDNESS PROPERTY STUDY FOR UHMWPE COMPOSITES USING NANO INDENTATION

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## **Abstract**

Ultra high molecular weight polyethylene (UHMWPE) is used as articulating surface material for hip joint replacements. Due to permanent deformation of material it leads to dislocation of artificial hip joint. Composite material was developed by blending UHMWPE with varying percentage of Multi walled carbon nanotubes reinforcement. This blended mixture of material is compressed in compression molding machine to get sheets. Hardness and Creep tests were carried on Composite specimens and the results were compared and validated using Oliver and Pharr analytical model. It is observed that, as the weight percentage of multiwall carbon nanotube is increased the hardness increases up to 25% and creep displacement decreases up to 29% when compared with pure Ultra high molecular weight polyethylene material

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### **Keywords:**

Creep;  
Hardness;  
NanoIndentation;  
UHMWPE;  
MWCNT.

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## **1. Introduction**

In the past, different materials are studied and reviewed for bearing surfaces applications. Biologically there are few reasons because of which hip joint fails and lead to hip joint replacement as explained by S. Ramakrishna et al [1]. George Matsoukas et al [2] stated liberation of wear particles at articulate surfaces of hip joint lead to inflammatory defined as Osteolysis. Also permanent deformation due to creep effect leads to dislocation of hip joint. The solution is to develop composite materials with improved Creep, Stiffness and Hardness properties. Ultra high molecular weight polyethylene material has excellent properties like high

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wear resistance, strength, chemical stability and bio compatibility. Thus it is used for Total hip replacement from many years [3-5].

Creep displacement of Ultra high molecular weight polyethylene is higher than conventional metals which introduce the gap between acetabula cup and femoral head leading to loosening of the hip joint and dislocation failure. Also, Wear debris liberation of Ultra high molecular weight polyethylene material lead to loosening of implants which in turn cause the long term failure of Total hip joint replacement [7]. Hence study is carried out to improve the creep, hardness and modulus properties of Ultra high molecular weight polyethylene by blending multi wall carbon Nano tube to increased life span of Total hip joint replacement.

The objective of the present study is to compare the creep displacement, hardness and reduced modulus property of pure Ultra high molecular weight polyethylene with multiwall carbon nanotube blended with Ultra high molecular weight polyethylene composite material. The essence of the present work is to get an idea regard the effect of Multi walled carbon nano tube (MWCNT) reinforcement variation on Creep and Hardness properties. Experimental results are validated by Oliver and Pharr analytical model.

## 2. PREPARATION OF COMPOSITE

Multi walled carbon nano tubes (MWCNT's) are used as filler material along with Ultra high molecular weight polyethylene for the preparation of composites sheet. Multi walled carbon nano tubes has the dimensions of outer diameter ranging from 8 to 15 nm, length 10 to 50  $\mu\text{m}$  having density of  $2100 \text{ Kg/m}^3$  ( $2.1 \text{ g/cm}^3$ ) and the purity was reported to be more than 95% with an ash content of 1.5%. Ultra-high molecular weight polyethylene with molecular weight ranging from  $3.5 \times 10^6$  to  $6 \times 10^6 \text{ g/mol}$  (ASTM calculation) is used as base material. Ultra-high molecular weight polyethylene will be in the form of powder with particle size ranging from 0 - 500 $\mu\text{m}$ , with an average size in the range of 135- 150  $\mu\text{m}$ . The density of Ultra-high molecular weight polyethylene powder is  $930 \text{ Kg/m}^3$ . Chemically treated Multi walled carbon nano tubes (MWCNTs) and Ultra-high molecular weight polyethylene (UHMWPE) was physically blended using Plastic corder machine shown in Figure-1 for 30 minutes. The temperature of the compound inside the chamber is maintained at  $200^\circ \text{C}$ . The "dough" obtained after melt-mixing the powder were put in the die. During compaction stage, temperature of the die plates was also maintained at  $200^\circ \text{C}$  and the material is first compressed to 19 MPa (190 bar) for one minute. The die is unloaded to allow recrystallization of the material at  $200^\circ \text{C}$ . Further the die is loaded again to attain a pressure of 19 MPa (190 bar) for five minutes. Machine used for this purpose is shown in Figure-2. Finally die is unloaded and water cooled to room temperature before the composite sheets are removed. The size of compression molded sheets is 150 x 150 x 2mm which is shown in Figure 3.



Figure 1. *Brabender Machine*



Figure 2. *Hot Press Machine*



Figure 3. *Compression moulded Ultra-High Molecular Weight Polyethylene sheet*

### 3. HARDNESS TEST

Hardness test is performed using Keysight Nano indenter G200 make. Tests were performed for UHMWPE, UHMWPE + 1% CNT, UHMWPE + 3% CNT and UHMWPE + 5% CNT materials. The Specimen is sized to dimension of 25mm x 25mm and test surface are cleaned. Nano indentation was performed using Berkovich diamond indenter. Test is carried out by applying load in milli Newton ranging from zero till the indentation depth reaches 3000nm and then the specimen is unloaded. As the load is applied, the depth of penetration is measured. The area of contact at full load is determined by the depth of the Impression and the known angle or radius of the indenter. The hardness is found by dividing the load by the area of contact. Shape of the unloading curve provides a measure of elastic modulus. Load versus displacement are recorded for each sample, when indenter reaches final indentation depth Hardness and modulus value is displayed.

#### 4. HARDNESS RESULTS AND DISCUSSION

Load applied and indentation depth is the output of the experiments conducted. Also equipment displays hardness and reduced modulus values directly. Load is gradually applied till the indentation depth reaches 3000nm and then the specimen is unloaded.

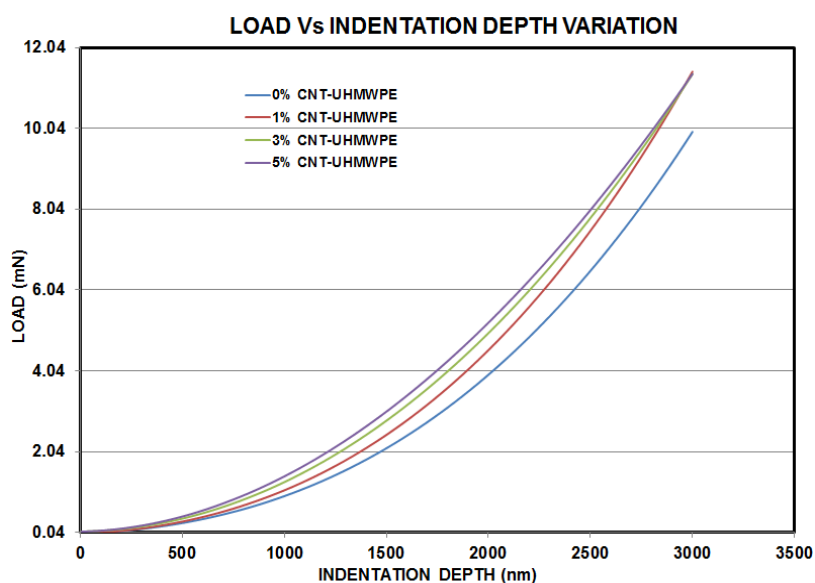


Figure 4. Load Vs Indentation Depth

Indentation depth variation with respect to load is shown in figure 4. From the figure it is observed that, the ascent of curve due to load application is increased with increase in MWCNT weight percentage in the polymer. This behaviour indicates that, to have same indentation depth the load required is more when MWCNT concentration is high in the polymer composite. Also it justify that the Ultra high molecular weight polyethylene material is hardened when the filler material MWCNT is added. To have indentation depth 2000nm, the load required for pure Ultra high molecular weight polyethylene is 6.5mN and with 1%, 3% and 5% filler addition the load required to have same indentation depth increases to 7.5mN, 7.82mN and 8mN

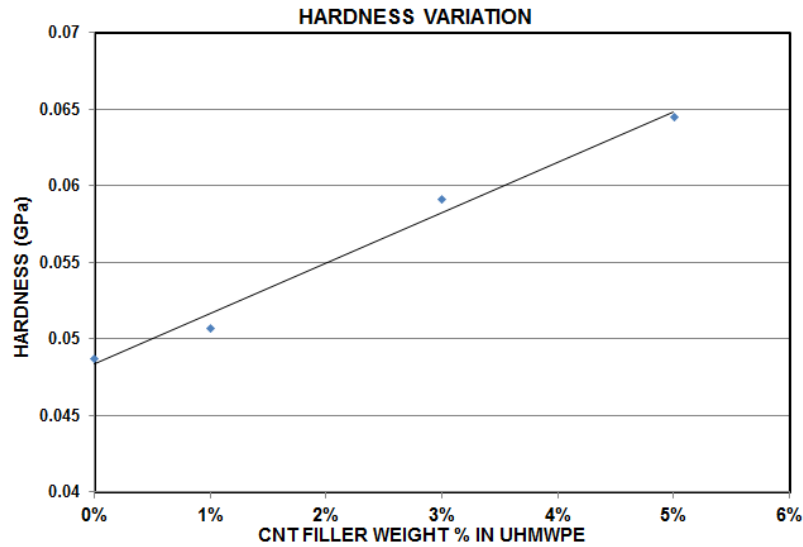


Figure 5. Hardness variation with respect to filler variation

Figure 5 shows Hardness variation with respect to filler material weight percentage. Hardness value quoted by test equipment is averaged to get single hardness property for respective material. It is observed that hardness value increases linearly with increase in carbon nano tube weight percentage in pure UHMWPE material. For pure UHMWPE material it is observed that hardness value is 48.7 MPa but when 5% carbon nano tube is added to polymer, hardness increased to 64.5 MPa.

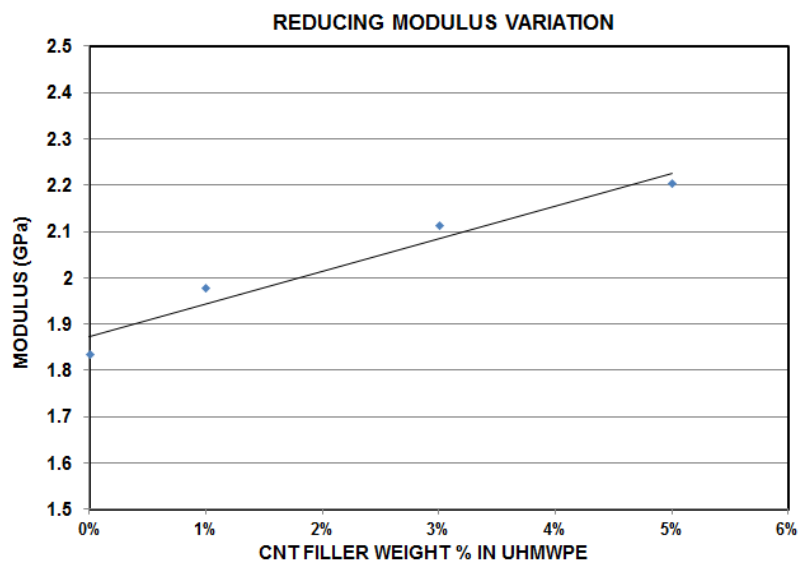


Figure 6. Reducing Modulus Variation

Figure 6 shows Modulus variation with respect to filler material weight percentage. It is observed that modulus value increases linearly with increase in carbon nano tube weight percentage in pure UHMWPE material. For pure UHMWPE material it is observed that hardness value is 1.8 GPa but when 5% carbon nano tube is added to polymer, modulus increased to 2.2 GPa.

MWCNT addition to UHMWPE will increase the toughness of the composite material which in turn makes material harder which is evident from discussed results

## VALIDATION

The experimental results are validated using the Oliver and Pharr analytical model. The indentation load (P) with displacement (h) data obtained during one full cycle of loading and unloading. The important parameter in the model is S, which has the dimensions of force per unit distance and is known as elastic contact stiffness. Hardness (H) of material and Modulus( $E_r$ ) property is evaluated using this parameter. The fundamental relations from which H and E are determined are:

$$H = P/A \quad \dots\dots\dots (1)$$

Where P is the load in milli N and A is the projected contact area at the corresponding load, and:

$$E_r = \frac{\sqrt{\pi}}{2\beta} \frac{S}{\sqrt{A}} \quad \dots\dots\dots (2)$$

Where  $E_r$  is the reduced elastic modulus and  $\beta$  is a constant that depends on the geometry of the indenter.

Using equation-1 hardness value for all samples are evaluated and plotted against experimental results as shown in Figure 7. The deviation in validation is approximately 13%. Tabel-1 shows hardness values obtained from experimental and analytical calculation.

Material	Hardness (Mpa)		Deviation (%)
	Experimental	Analytical	
Pure UHMWPE	48.68	42.31	13
1% MWCNT	50.75	44.55	12
3% MWCNT	59.17	50.90	14
5% MWCNT	64.47	55.26	14

Table 1. Hardness values and deviation

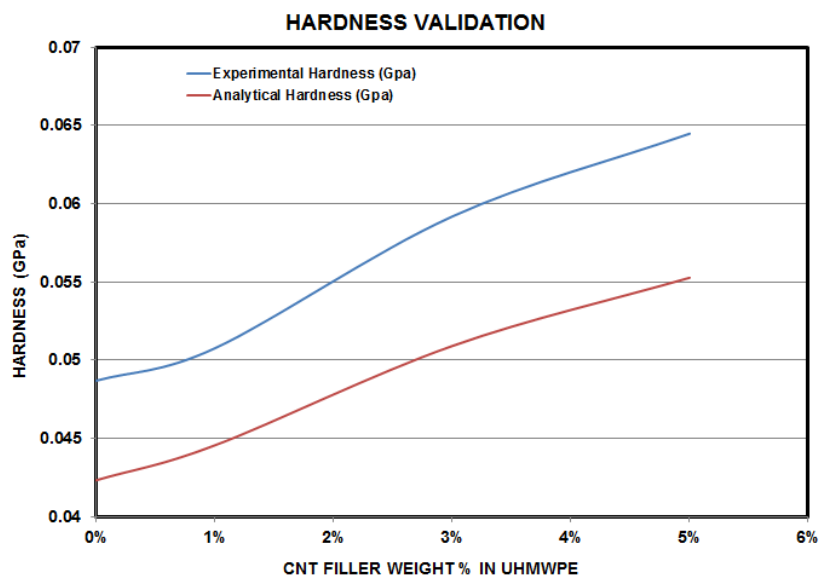


Figure 7. Hardness validation

Similarly reduced modulus is evaluated analytically for 5% CNT composite which is compared against experimental results as shown in Figure 8. The deviation in validation observed is 25%. Though the deviation is observed in validation the deviation remains constant for all conditions.

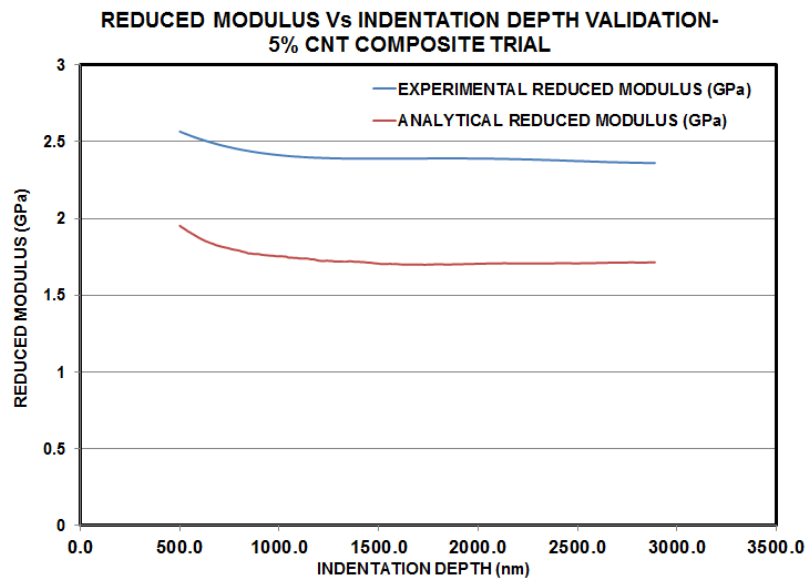


Figure 8. *Reduced Modulus validation*

## 5. CREEP TEST

Creep test is performed using Keysight Nano indenter G200 make. Testing parameter and indenter type considered is similar to what used in hardness testing. To simulate creep behavior, the sample is made to undergo constant loading for standard duration once it reaches stated indentation depth. Testing procedure as explained below.

Test is started by applying load in milli Newton ranging from zero till the indentation depth reaches 2300nm and then the specimen is continued to load at constant value of load for 10mins. Load versus displacement are recorded for each sample. When load is held constant for 10 mins, displacements are tabulated to obtain creep displacement with respect to time. Also creep strain percentage is evaluated using initial indentation depth and indentation depth after 10mins of constant loading. These creep properties are compared with all four configuration of materials.

## 6. CREEP RESULTS AND DISCUSSION

Load applied and indentation depth is the output of the experiments conducted. Using this data creep displacement with respect to time is extracted.

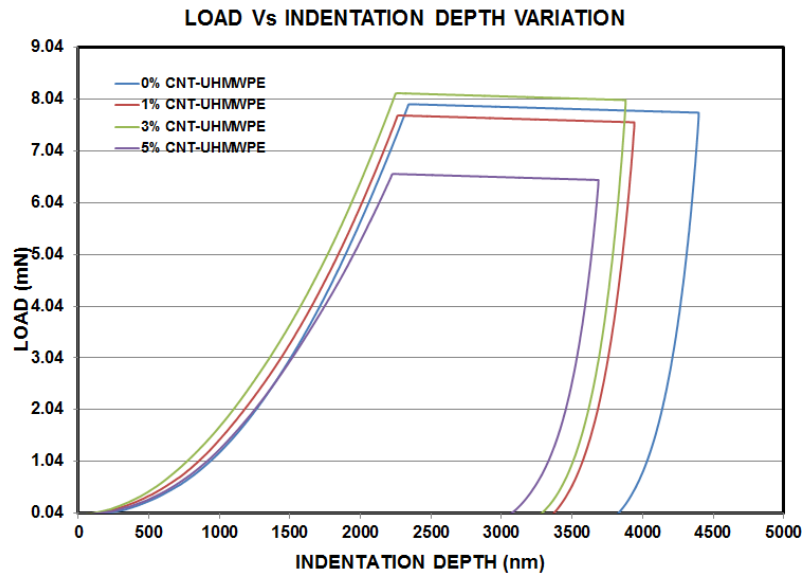


Figure 9. Load Vs Indentation

Figure 9 shows load variation with respect to indentation depth for all the materials which is the output of the experiments. Compared to hardness test, here after loading the sample up to predefined indentation depth the samples undergoes constant load for constant defined time that is 10mins and then unloaded. From the results it is observed that the creep displacement after 10mins is not same for all the materials. By appearance pure UHMWPE experiences more creep displacement and 5% MWCNT reinforced polymer composite experiences least creep displacement

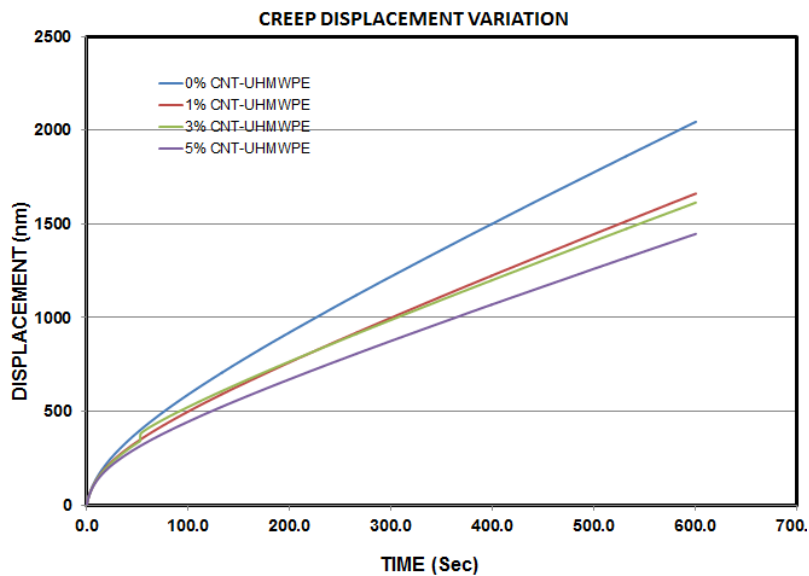


Figure 10. Creep Displacement wrt time

Figure 10 shows creep displacement variation with respect to time for all the materials. The displacement experienced for constant load condition at fixed time period is used to extract creep displacement variation with respect to time. It is observed that creep displacement increases as the time increases. The point at which indentation depth 2300nm is reached and after that the load is kept constant is considered as zero time zero displacement. To quantify after 10mins, pure UHMWPE material experiences creep displacement of



2045nm. Whereas composite material with 1%, 3% and 5% weight percentage of MWCNT experiences 1662nm, 1613nm and 1446nm of creep displacement.

Figure 11 shows creep strain variation with respect to time for all the materials. To evaluate creep strain indentation depth of 2300nm is considered as actual penetration and indentation after 10mins of hold time is considered as change in penetration. It is observed that creep strain reduces linearly as MWCNT weight percentage increases in UHMWPE composite material.

The creep resistance of material increases as Carbon Nano tube is reinforced with Ultra high molecular weight polyethylene. It can be studied that creep strain value decreased from 87% for pure polymer to 72.8% for 1 Wt. % composite samples which lead to 14% decrease. The creep strain of 3 Wt. % and 5 Wt. % composite samples decreases to 71% and 64%.

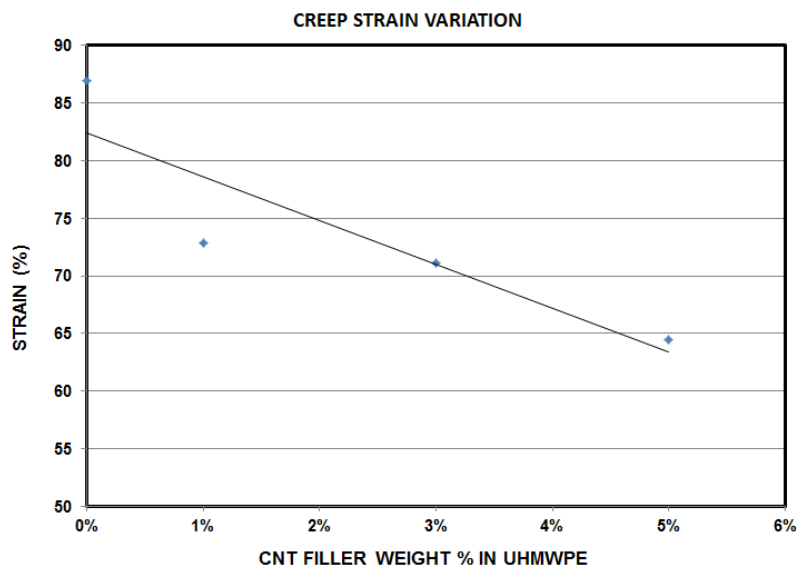


Figure 11. Creep strain variation

From the results it is understood that creep resistance increases as filler material MWCNT is added to pure UHMWPE material. The MWCNT has very good toughness property which in turn helps in enhancing the hardness and creep resistance property.

## 7. CONCLUSION

In this article the Ultra high molecular weight polyethylene and the Ultra high molecular weight polyethylene composites are successfully fabricated using compression molding technique and the effect of Carbon nano tube weight percentage is studied. The conclusions are as follows:

1. CNT reinforced composites shows improved creep properties compared to polymer alone material. With 1%, 3% and 5% CNT addition to polymer the creep displacement decreased by 18%, 21% and 29%

2. CNT reinforced composites shows improved surface hardness value compared polymer alone material. With 1%, 3% and 5% CNT addition to polymer the hardness value increased by 4%, 18% and 25%
3. Elastic modulus increases as carbon nano tube added to polymer alone material. With 1%, 3% and 5% CNT addition to polymer the modulus value increased by 7%, 13% and 16.7%
4. Using analytical model, Experimental results for Hardness value are validated with 13% variation and Modulus value are validated with 24 % variation

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