Thermo-mechanical behaviour of ultrahigh molecular weight polyethylene-carbon nanotubes composites under different cooling techniques

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Abstract. Ultrahigh molecular weight polyethylene (UHMWPE) is a unique polymer with outstanding physical and mechanical properties that makes it particularly attractive to fabricate the bearing surface for artificial joints. The growing demand for UHMWPE as a bearing material for joint replacement has lead to active research in the development and understanding of its properties in addition to nanocomposites which is being received a considerable attention by industries. Despite the requirement of visco-elastic properties of the UHWMPE and its composites, the characterization of them has received relatively little attention. The objective of this work is concerned with the studies on visco-elastic behaviour of UHMWPE and nanocomposites, which were prepared at optimized ball milling time with different cooling techniques. It is observed that stiffness of the materials increases appreciably at 0.2wt.% CNTs with an increase of frequency till 30Hz which confirms the reinforcing effect of CNTs in composites. The loss modulus of the sample is observed to be converged at higher temperatures irrespective of frequency and the damping effect of the sample could be kept within the limit of polymer at any frequency range when the temperature is low and it is also possible at any temperatures at higher frequencies except LN2 cooled sample. The relaxation fraction increases with an increase of temperature and decreases with an increase of frequency. It is concluded that air cooled sample could be used wherever modulus is the main criteria irrespective of temperature and frequency, LN2 cooled sample can be used where more damping is required and water cooled samples may be used where more strength and toughness are required.

Introduction

Ultra-high molecular weight polyethylene (UHMWPE) has found a wide application as a load bearing material in the majority of joint prostheses in combination with metal or ceramic counterparts. Although the short-term function of UHMWPE implants is found to be satisfactory, their long-term performance has been limited due to wear of the implants. Due to such concerns, the scientific literatures contain extensive information dealing with improving wear behaviour as well as the mechanical properties of UHMWPE using different techniques. An attempt is being made to increase the properties of UHMWPE by incorporating carbon nanotubes (CNTs) to prepare nanocomposites. Compared with other composites and polymers, the nanocomposites generally exhibit an outstanding improvement in their mechanical properties and thermal stability, even at a
very low filler concentration [1-3]. Despite the fact that the in-vivo environment of UHMWPE implants includes cyclic loading, elevated temperature, and varying frequency conditions are expected to play an importance role in clinical performance of artificial joints, the experimental and analytical characterization of the visco-elastic properties of UHMWPE and its improvement has received relatively little attention. A better understanding of the dynamic mechanical properties of the composite will help to define structure/property relationships as it is sensitive to internal structure and subsequently to relate these properties to the product final performance, Kaynak et al. [1996]. Several studies have been carried out on the DMA of fibre reinforced and particulate filled composites to investigate the effect of addition of fillers, coupling agents, etc., on fibre matrix interface. Though it is known that reinforcements are purely elastic systems while the polymer and the CNT/polymer interface are viscoelastic, limited work has been performed previously relating to visco-elastic behaviour of UHMWPE-CNT composites. Because of the time-temperature equivalence in viscoelastic materials, the dynamic mechanical behaviour may be tested against frequency and temperature, when the effect of longer time (lower frequency) is equivalent to that of higher temperature which provides an insight into the level of interactions between the polymer matrix and CNTs reinforcement.

Review

Many researchers have employed dynamic mechanical analysis in characterizing the interfacial properties of the composite materials. Wielage et al. [2003] observed that the quality of the composite material such as matrix-fibre adhesion and damping behaviour could be determined from the DMA study. Alvarez et al. [2003] studied viscous properties of composite material by varying the influencing parameters and observed that aspect ratio of the sample should be higher than 15 and the heating velocity equal to 5°C/min to obtain very good results on DMA. Shaffer et al. [1999] performed DMA analysis on carbon nanotube/poly (vinyl alcohol) composites and they showed that the elastic modulus of the material is significantly increased by the presence of carbon nanotubes. Kanagaraj et al. [2008] observed that the storage modulus of the HDPE increases with an increase of CNTs. Gerard et al. [1990] concluded that the storage modulus of the materials was decreased due to increased mobility of polymer chains in composites having poor adhesion between polymer and reinforcement. Kontou et al. [2006] attempted theoretical and experimental studies on viscoelastic beaviour of unidirectional epoxy-vinyl resin-glass fibre composites. Khatibi et al. [2002] studied the existence of different interphases in the composites using DMA and found that cyclic loading affects the fibre-matrix interfacial properties in the composites. Tsagaropoulos et al. [1995] suggested that DMA can be used to measure the chain mobility which occurred due to polymer-filler interaction. DMA has also proved to be useful in studying the interfacial properties of laminated epoxy–glass composite samples, Thomasan [1995]. Fouad et al. [2005] observed that pre-heat treatment process on UHMWPE strongly affects its mechanical and visco-elastic properties. Tajvidi et al. [2004] observed that the temperature dependence of shift factor of Kenaf fibre/HDPE composites was found to be conform to an Arrhenius equation whereas Deng et al. [1998] confirmed its applicability in UHMWPE. Yang et al. [2007] studied thermo-mechanical properties of HDPE-VGCNF composites by DMA and observed that both storage and loss modulus of the composites increase with an increase of draw ratio. Nazhat et al. [2000] observed that an increase in hydroxyapatite (HA) concentration increased the storage modulus of polyethylene-HA composites at all temperature ranges and reduced storage modulus at all volume fraction of HA with an increase of temperature. They also found that the smaller particle size of HA in polyethylene works more efficiently than bigger size particles. Jin et al. [2001] observed that storage modulus of CNT-PMMA composites is increased by the incorporation of nanotubes at high temperature. Blaker et al. [2004] concluded from their DMA studies on commercially available sutures that the silver doped bioactive coating caused no significant effect on the transition temperature and visco-elastic behaviour compared with uncoated suture. As the thermal dependence of intermolecular
cooperativity based on the Williams–Landel–Ferry (WLF) equation yields more quantitative analysis of viscoelastic properties, Mäder et al. [2007] used cooperativity to model the viscoelastic response of glass fiber/epoxy resin composites.

**Materials and methods**

**Materials**

The medical grade UHMWPE was supplied by M/s Ticona, Inc, Germany. The trade name of the material used in this study was GUR 1020. The specifications of UHMWPE powder are as follows: density 930 kg/m³, mean particle size 140 µm and the average molecular weight 3.5 million g/mol. The multi walled carbon nanotubes (CNTs) were purchased from M/s Shenzhen Nanotech Port Co., Ltd., China. The specifications of CNTs are as follows: range of diameter 60–100 nm, length of the tubes 5–15 µm, and purity >95%.

**Sample preparation**

The required quantity of CNTs was suspended in the mixture of concentrated nitric acid and sulphuric acid by the volume ratio of 1:3 and refluxed at 140°C for 30 min. After washing the nanotubes with distilled water until the supernatant attained a pH around 7, the samples were dried at 100°C and thus the chemically treated CNTs were obtained. The main purpose of this process is to remove amorphous carbon, impurities, metal catalyst residue and attaching functionalised groups on the surface of CNTs to allow chemical bonding with polymer while preparing composites. Though different attempts have been carried out to prepare homogeneous dispersion of CNTs in UHMWPE, physical blending method was chosen in this study. The chemically treated CNTs and UHMWPE powder was ball milled for 45min. using ceramic balls at room temperature to obtain the homogeneous mixture of raw material, which was filled in the mould cavity in order to obtain a compressed sheet. During the compaction stage, the temperature of the mould plates was adjusted to 200°C and the raw material was first compressed at 13.8 MPa for 60 sec. Then, the mould was further unloaded and loaded for 4-5 times at an interval of 30 s followed by a sintering stage at 200°C for 5 min under a load of 6.9 MPa. During the following recrystallization process at 200°C, the mould was once again loaded at 13.8 MPa for 5 min. Then, the mould was unloaded and removed from the heated plates and cooled by air, water and liquid nitrogen (LN2). Thus, the compression moulded sheet was prepared and the test sample for DMA studies was cut from it. It is observed from our previous studies that the optimized milling time for UHMWPE and CNTs mixture was to be 45min. of milling time, Kanagaraj et al. [2008], where the least quantity of CNTs i.e. 0.2wt.% in UHMWPE was chosen. The objective of this work is concerned with the studies on visco-elastic behaviour of the UHMWPE and nanocomposites, which were prepared at optimized ball milling time with different cooling techniques.

**Experimental work**

DMA experiments were carried out using Perkin-Elmer (DMA 7) equipment and the samples were cut from the compressed sheets of polymer and composites. The principle of DMA test is to apply a sinusoidal extensional strain to the test sample while simultaneously measuring the stress in the sample over a range of temperature and frequency. The relative amplitude of the stress and strain and phase shift between the two signals were then used to determine the storage modulus, loss modulus and tan δ as a function of temperature and frequency. This procedure was repeated on each sample at 11 different frequencies varied from 0.1Hz to 50Hz over the temperature range of 27°C - 62°C at an interval of 5°C where the reference temperature was kept at 37°C. Only, the averaged data were plotted without error bars in order to present the results clearly.

**Results**
Storage modulus
It is observed from Fig.1 that storage modulus was increased from 708 MPa to 908 MPa at 27°C and 0.2 wt.% CNTs in UHMWPE at 0.1Hz when the sample prepared by air cooling technique. It increases with an increase of frequency till 30Hz and then it starts to decrease, irrespective of the cooling technique used. Though 0.2wt.% CNTs used in UHMWPE, the cooling technique plays an important role to increase the storage modulus of the composites by increasing the crystallinity in the composites. The effect of air cooling technique on the sample is disguisable at all frequency and temperature ranges tested in this study compared with other samples. The increase of storage modulus for air cooled sample at 27°C is observed to be about 23-28% and it decreases with an increase of frequency within this limit. However, this increment increases with an increase of temperature irrespective of frequency. In case of water cooled sample, the increase of storage modulus is observed to be 4% compared with UHMWPE sample at 27°C and it is restricted within 2% at above 42°C irrespective of frequency and temperature. In case of LN2 cooling, the increment is within 16% at 27°C and it is reduced to 4% at 57°C. The drastic decrease of storage modulus in all samples is observed at 57°C. It decreases by more than 65% irrespective of cooling technique at 0.1Hz however it could be restricted within 50% with an increase of frequency till 50Hz. Except air cooled composite sample, the storage modulus of composites and polymer sample is inseparable at 57°C which diminishes the effect of CNTs and the cooling techniques. Fig.2 shows the variation of storage modulus with an increase of temperature at 0.1 and 50Hz. Only air cooled sample shows thermal stability of storage modulus and linear decrement with an increase of temperature at the tested frequency ranges. This sample even maintains about 30% increase of modulus compared to that of pure UHMWPE sample at 62°C irrespective of frequency. In case of LN2 cooled sample, storage modulus agrees within 3% with that of UHMWPE from 47°C onwards at 0.1Hz. However, the difference between them increases with an increase of frequency and temperature. In case of water cooled sample, it maintains about 5% increase of storage modulus at 27°C and 32°C. When the temperature increases beyond these temperatures, the effect of CNTs, temperatures and frequencies are not having any influence in the composites and its value is approximately equal to that of polymer. It can be observed that stiffness of the materials increase appreciably at 0.2wt.% CNTs with an increase of frequency till 30Hz and decreases with an increase of frequency.

Loss modulus
Fig.3 shows the effect of temperature, frequency and cooling technique on loss modulus (E´´) of the composites and UHMWPE. In general, E´´ of the test sample at 27°C decreases linearly till 3Hz and further reduces to 30Hz by polynomial path where a reversing trend on the effect of frequency was observed. It is observed to increase with an addition of CNTs and type of cooling techniques used. Some interesting feature on LN2 cooled sample is that E´´ of this sample is more than any sample tested in this study at 27°C. However, with an increase of temperature, E´´ of the sample is initially lesser than air cooled sample till transition frequency beyond which reverse trend was observed. The transition frequency increases with an increase of temperature of the test sample. It is also observed that the trend of E´´ is changed with an increase of temperature. At 57°C, E´´ of the sample remains constant till 1Hz and then linearly decreases till 30Hz where it is started to increase. The percentage increase of E´´ of the sample increased with an increase of frequency. Fig. 4 shows the variation of loss modulus with an increase of temperature at 0.1, 1 and 10Hz. It is observed that α relaxation temperature (Tα) of test sample increases with an increase of frequency till 10Hz, which is listed in Table 1. However, above which Tα was not observed in the test range of the temperature where a linear increase of loss modulus against temperature was observed with an increase of frequency. It is also observed that E´´ of the sample is observed to be converged at higher temperatures irrespective of frequency.

Damping factor (Mechanical relaxation process)
The mechanical relaxation process that occurs in the test sample at different temperatures and frequencies is shown in Fig.5. The mechanical relaxation increased with an increase of temperatures. It is observed that LN2 cooled sample is having more mechanical relaxation than other composites and polymer at all frequency and temperature range at which the samples were tested. It increases with an increase of temperature at any frequency and reaches its maximum at 62ºC and it is observed to be 80% at 0.1Hz and 71% at 50Hz. The damping effect of the sample could be kept within the limit of polymer at any frequency range when the temperature is low and it is also possible at any temperatures at higher frequencies except LN2 cooled sample. In case of air cooled sample, it has the lowest mechanical relaxation among composites and polymer samples at the testing range of and temperatures and frequencies except at 50Hz, where it has more relaxation than pure polymer. In case of water cooled sample, the mechanical relaxation is within 11% compared to that of polymer in all temperature and frequency ranges. The variation of mechanical relaxation against frequencies at three different temperatures is shown in Fig.6. It is observed that tan δ decreases with an increase of frequency till 30Hz and above which opposite trend was observed. The mechanical relaxation of air cooled sample at 27ºC is maintained less than 12% as that of UHMWPE till 30Hz and above which relaxation turn to be in opposite trend i.e. 27% more than that of UHMWPE. At 42ºC, the relaxation fraction is less than 7% as that of UHMWPE at lower range of frequency. However, at 50Hz, the relaxation fraction varies between 21-28% more than that of UHMWPE in the temperature range of 27 to 62ºC and it decreases with an increase of temperature. The same trend is observed at 57ºC as well, where relaxation fraction till 30Hz is found to be within 3% where as at 50Hz, it is observed to be 22%. In case of LN2 cooled sample, the relaxation fraction at 27ºC is observed to be within 5% till 30Hz where as it is observed to be 54% at 50Hz. The maximum relaxation at 62ºC is 80% at 0.1Hz and it starts to decrease till 30Hz and then increases to about 70% at 50Hz. In general, the relaxation fraction increases with an increase of temperature and decreases with an increase of frequency.

Discussion

Since UHMWPE is a viscoelastic material, it shows both the capacity to store mechanical energy without dissipation of it and the ability to dissipate energy but not to store it. The dissipation of energy manifests itself as an internal friction or damping in a polymeric material. In a polymer nanocomposite, the energy dissipation may also come from the CNT-matrix interface where friction between the two phases can occur. The internal friction can be quantified by tan δ which is the ratio of the energy dissipated per cycle to the energy stored during the cycle. A high damping capability is essential to reduce the effect of undesirable vibrations by decreasing the amplitude of resonance vibrations within safe limits. But a high damping capability is also accompanied by a decrease in dimensional stability. It appears that the addition of CNT has actually increased the mobility of the amorphous phase in the polymer, and hence it increased the damping of the composite. In the dissipation of energy during DMA, a relatively large amount of the energy is assumed to be dissipated at the CNT-UHMWPE interface due to the interfacial bond, Wang et al. [1998].

The storage modulus reveals the capability of a material to store mechanical energy and resist deformation. Ni et al. [2002]. The temperature curves of storage modulus and loss factor reveal the differences between pure UHMWPE and composites. The storage modulus reaches higher values with an addition of CNTs and the same effect was observed in case of loss modulus. Due to semicrystalline nature of UHMWPE, an addition of nanotubes does not produce a dramatic increase in the stiffness of the polymer matrix. However, it is evident from the experimental results that there was a notable increase in the storage modulus of UHMWPE with the incorporation of CNTs. This is probably due to increase in the stiffness of the matrix with the reinforcing effect imparted by the CNT that allowed a greater degree of stress transfer at the interface, George et al. [1996]. This behavior is primarily attributed to improved interfacial adhesion between the CNT and UHMWPE. The storage modulus of the nanocomposite increased monotonously with an addition of CNTs till 30Hz, indicating that CNTs have a strong reinforcing effect on the elastic properties of the matrix which shows a strong dependence of the visco-elastic behaviour of UHMWPE on the test frequency.
and strain rate dependent. There are different parameters affecting the molecular motion within the matrix. Thus, it would be expected that polymer composites with different cooling techniques exhibit different modulus enhancement at different temperature ranges. It is observed from the experimental results that the storage modulus of the tested samples decreased with an increase of temperature. However, the rate of reduction of the modulus was compensated by the interactions caused in the presence of CNTs in the nanocomposites, which further shows an increase in thermal stability of the matrix with the addition of CNTs, in case of air cooled sample. The modulus of the composites changes with temperature as the molecular motions within the polymer changes. The storage modulus increased by the stiffening effect of the nanotubes which is particularly significantly at lower temperature. The enhancement of storage modulus reduces drastically with an increase of temperature for water cooled and LN2 cooled sample whereas air cooled sample is showing exception which maintains the same enhancement of storage modulus even at higher temperature.

The loss modulus represents the viscous components of the sample and is equivalent to the energy dissipated through deformation. When the composite was subjected to external stresses, energy was dissipated by the CNT-Polymer interaction and the friction between CNT-CNT. The peak of relaxation transition is observed at different temperatures with an increase of frequency which is related to a complex multi relaxation process and mainly concerned with the molecular motion of the crystalline region of polyethylene [Huang et al. 2004]. The displacement of higher peak is observed with an increase of frequency. The $\alpha$ relaxation is indicated by the peak in the loss modulus curves occur in the temperatures range of 27 to 60ºC, which increases with an increase of frequency and is due to the increased crystallinity as a result of different cooling techniques. These results agree with Pegoretti et al. [2000] and Fouad et al. [2005], which showed that the $\alpha$ relaxation of different types of polyethylene and UHMWPE occurs at a temperature range depending on the degree of crystallinity. The present results show that the loss modulus changes significantly due to an addition of CNTs but decreases with an increase of frequency.

The damping factor of the materials is an indication of the fractional energy lost in a system due to deformation and it gives the balance between the elastic phase and viscous phase in a polymeric structure. When the temperature increases, the thermal energy given to the polymer chains also increases allowing greater energy loss and thus the damping factor increases with an increase of temperatures. However, energy loss mechanism is very complex in nanocomposites with the possibility of additional losses occurring at the CNT-polymer interface. A decrease in damping factor with an increase of frequency is associated with the improvement of interfaces in CNT-matrix systems. When the damping is higher, it can be attributed to the increased mobility of the polymer chains in composites with poor adhesion. The area under the temperature-tan $\delta$ graph can be used to quantify the energy dissipation by each material system. From the experimental results, it is observed that LN2 cooled sample is having more damping than any other samples and the composites show a higher damping than pure UHMWPE, which is due to the visco-elastic energy dissipation as a result of CNT-CNT friction and CNT-UHMWPE interaction, Huang et al. [2004].

**Using DMA Data for WLF Equation**

The commonly used empirical equation for time–temperature superposition (TTS) is the Williams–Landel–Ferry (WLF) equation, which relates a shift in temperature with a shift in time, Ferry [1980], with an assumption that the free volume increases in linear way with increasing temperature. It is very useful if the information is available for only one temperature and the information must be computed for other temperatures. The WLF-equation is primarily used when the time–temperature behaviour of a polymer in the area of the glass transition has to be described. The WLF equation is used to describe the complete temperature and concentration dependence of the viscous molecular relaxation time or viscosity and it is mathematically described by:
\[
\log_{10}(a_T) = \frac{-C_1(T - T_0)}{C_2 + (T - T_0)}.
\]

where \(a_T\) - shift factor, \(C_1\) and \(C_2\) - constants, \(T_0\) - reference temperature (K) and \(T\) - temperature at which shift factor is calculated. Fig.7 shows the variation of WLF factor with an increase of temperature for the tested samples and it is observed that all test samples follow the same trend and they are inseparable each other. The experimental results and the calculated values from WLF model follow second order polynomial equation. The coefficients of the WLF equations are given in Table 1. Comparing the calculated shift factor, it clearly confirms the choice of WLF model and it shows that the activation energy in the un-reinforced system corresponds to that of the reinforced composites. The relaxation processes of the UHMWPE system is maintained the same with nanocomposite system. Although the TTS technique was originally developed for amorphous polymers, Nielsen et al. [1994] suggested that it could be applied to semicrystalline polymers as well. A plot of \(\log a_T\) against the reciprocal of the absolute temperature gave a linear relation, indicating an Arrhenius relationship.

**Mechanical properties**

In order to compare the results obtained from the DMA studies, the mechanical properties of the test sample was studied using a standard Universal testing machine. It is observed that air cooled sample has the maximum enhancement of Young’s modulus i.e. 27% compared to that of polymer, which supports the results of storage modulus obtained from this study. However, in case of tensile strength, toughness and breaking elongation, the enhancement is observed to be 22.7%, 34.8% and 26.3%, respectively for water cooled sample, Kanagaraj et al. [2000]. The mechanical properties of the sample are also quoted in Table 1.

**Limitation and scope**

The thermo-mechanical characterization of composites prepared with different cooling techniques was studied at unique concentration of carbon nanotubes and compared their results with pure polymer. More experimental studies on UHMWPE-CNTs composites are in progress in order to evaluate the effect of the concentration of CNTs in UHMWPE matrix on the thermo-mechanical properties of the nanocomposite.

**Conclusions**

The following conclusions are derived from the above study.
1. Air cooled composite has good thermal stability of storage modulus with an increase of temperature and frequency compared to that pure polymer and other composite samples.
2. When strength and toughness of the materials are considered, water cooled sample offer best results than other samples.
3. Dual increase of storage modulus and loss modulus of the sample by the addition of CNTs in UHMWPE.
4. LN2 cooled composites show higher damping than pure polymer and composites.
5. The reinforcing effect of nanotubes on the composite was confirmed from this study where a noticeable increase in the storage modulus was observed.
6. The visco-elastic properties of UHMWPE are sensitive to loading frequency and temperatures.
7. These results could be used to analyze the effects of frequency and temperature on the dynamic response of UHMWPE and nanocomposites under in vivo conditions.
8. The damping of the sample could be kept within the limit of polymer at any frequency range when the temperature is low and it is also possible at any temperatures at higher frequencies except LN2 cooled sample.
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References


Fig. 1. Storage modulus of UHMWPE and nanocomposites against frequencies

Fig. 2. Storage modulus of UHMWPE and nanocomposites against temperatures
Fig. 3. Loss modulus of UHMWPE and nanocomposites against frequencies.

Fig. 4. Storage modulus of UHMWPE and nanocomposites against temperatures.
Fig. 5. Damping factor of UHMWPE and nanocomposites against frequencies

Fig. 6. Damping factor of UHMWPE and nanocomposites against temperatures
Fig. 7. Shift factor of UHMWPE and nanocomposites against reciprocal of absolute temperatures.

Table 1. Properties of UHMWPE-CNT composites.

<table>
<thead>
<tr>
<th>Cooling technique</th>
<th>WLF equations constants</th>
<th>α relaxation temperature (°C)</th>
<th>Mechanical properties</th>
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<tbody>
<tr>
<td></td>
<td>C1</td>
<td>C2</td>
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<tr>
<td>Air</td>
<td>-15.163</td>
<td>114.58</td>
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<tr>
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<td>156.38</td>
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<tr>
<td>UHMWPE</td>
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<td>27</td>
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