

Wear resistance of graphene reinforced ultra-high molecular weight polyethylene nanocomposites prepared by octa-screw extrusion process

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ABSTRACT

Ultra-high molecular weight polyethylene (UHMWPE) is one of the well-established bio-materials for artificial prostheses. In order to improve its wear resistance, graphene incorporated UHMWPE nanocomposites are synthesized by octa-screw extrusion process. The exceptional compounding ability of the process is confirmed by uniform graphene dispersion in the matrix. An 18% improvement in yield strength, superior anti-wear property, 67% increase in crystallinity, and 4% increase in the decomposition temperature as compared to pure UHMWPE is measured. The nanocomposites are also more hydrophilic and show absence of cytotoxicity. The UHMWPE/graphene nanocomposites prepared by octa-screw extrusion are a potential candidate for joint replacement applications.

1. Introduction

In the past few decades, ultra-high molecular weight polyethylene (UHMWPE) has been widely used in orthopaedic implants during joint replacement procedure, due to its unique features such as high mechanical strength, bio-compatibility, chemical stability, low friction and electrical insulation [1]. Despite these advantages, the wear resistance of the material requires significant improvement to support prolonged use [2]. The wear resistance shall be enhanced by gamma and electron beam radiation exposure. However, other mechanical properties such as flexibility and fatigue resistance are deteriorated by the radiation treatments. One of the solution to overcome these concerns is to develop new materials that have improved wear resistance which are capable of extending the implant lifespan. In particular, the incorporation of suitable materials into the polymeric matrix to enhance the performance is a potential option [3]. Carbon based nanoscale materials such as carbon nanofibers, carbon nanotubes, and graphene [4], are 1D and 2D carbonaceous materials that have extensively gained recent attention. Previous studies have confirmed improved tribological properties of UHMWPE nanocomposites that are incorporated with suitable reinforcements such as carbon nanotubes and graphene [5–9]. The studies have demonstrated that the nanofillers aid in the improvement of the wear resistance in spite of their poor dispersion in the UHMWPE matrix which is highly viscous and possesses high molecular weight [10].

Among the different carbon-based compounds, graphene has been marked as a revolutionary material, mainly due to its excellent mechanical properties [4]. Graphene is a versatile 2D carbon nanomaterial that possesses a ultrahigh specific surface area, Young's modulus in the order of TPa and ultimate tensile strength of 130 GPa.

A good dispersion of nanofillers in the polymeric matrix is essential to overcome the adverse effects caused by their agglomeration. In order to effectively substitute graphene as a filler in polyethylene matrix, optimizing its composition and the method of preparation play a critical role. Different methods for dispersing the fillers such as ultrasonication [11], mini-lab extruder [12] and ball milling [13] have been employed to achieve a good mixing and distribution of the nanofiller in the UHMWPE matrix. Nevertheless, the experimental results have suggested little or no enhancement in the structural properties when compared to virgin UHMWPE. Alternatively, Yousef et al. [14] have investigated the addition of paraffin oil into carbon nanofillers reinforced UHMWPE and have reported that the additive decreases the viscosity, favors processability and supports uniform dispersion of the fillers in the polymeric matrix. Although the addition of paraffin oil or organic solvents facilitates improvement in the distribution of fillers in the UHMWPE matrix, this method is process intensive and the paraffin oil/solvents need to be extracted to prevent deterioration in the mechanical and wear properties.

The compounding of UHMWPE composites is a challenge, primarily

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due to the high molecular weight of the polymeric matrix which leads to exceedingly high melt viscosity and lack of melt flowability. In addition, most commercial UHMWPEs are available as powders, while most of the commercial extruding methods are usually designed to process pellets. This in turn makes the compounding of UHMWPE composites by traditional extruding processes difficult to accomplish [15]. Octa screw kneading method [16] is a fully inter-meshed co-rotating process with a greater number of kneading screws than the twin screw extruder. Hence the method offers excellent mixing and compounding capability. Furthermore, the octa-screw melt kneading method offers advantages such as reduction of heat generation during compounding and improved dispersion of fillers due to high-speed screw rotation. To the best of our knowledge, no previous research study has developed graphene filled solvent-free UHMWPE nanocomposites using octa-screw extrusion.

In this work, we have investigated the blending of UHMWPE nanocomposites with two types of graphene fillers using octa-screw kneader extrusion process. The morphology of the prepared nanocomposites has been characterized by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The thermal, mechanical and anti-wear properties are investigated. The cytotoxicity of the composites is also examined using cell culture assay.

2. Experimental

2.1. Materials

The polymers utilized in the study were Grade 320MU ultrahigh molecular weight polyethylene (UHMWPE), purchased from Mitsui Chemicals, Inc., Japan. The average molecular weight and the density of the as procured polymer was 3.2×10^6 g/mol and 935 kg/m^3 respectively. The two variants of graphene utilized in this study were G-11 powders with a thickness of 8 nm (about 20–30 layers) and a particle size less than $5 \mu\text{m}$, and, G-13 powders with a thickness of 60 nm (150–180 layers) and a particle size less than $7 \mu\text{m}$. Both the types of graphene powders were purchased from EM Japan, Japan.

2.2. Preparation of UHMWPE/Graphene nanocomposites

UHMWPE powder was kneaded using the two types of graphene powders with varying weight percentages, namely 0.1, 0.5, 1.0, 1.5 wt %, by octa-screw kneading extrusion process (WDR 150T-45MG-NH, Technovel Co., Ltd., Japan). In order to improve the dispersion, UHMWPE and graphene were initially dry mixed for 10 min. Subsequently, the mixed powders were placed in the octa-screw kneading extruder to produce UHMWPE/graphene strips. The temperature in the feeding side of the five cylinders was set as 180, 220, 240, 240, and $240 \text{ }^\circ\text{C}$ respectively. Further the die head and the extrusion die temperatures were set as $240 \text{ }^\circ\text{C}$ each as shown in Fig. 1. The kneading time

was 150 s, while the screw rotational speed was set at 400 rpm. The prepared strips were cut into small pellets of about 3 mm long which were subsequently ground into micrometer-sized powders.

Sheets of UHMWPE/graphene nanocomposites with a thickness of 0.2 mm were prepared by vacuum hot compression molding technique (Imoto machinery Co., Ltd). Owing to the polymer's high viscosity, the preparation procedure was completed in two steps. Firstly, the UHMWPE/graphene nanocomposite powders were hot pressed at $230 \text{ }^\circ\text{C}$ for 5 min under vacuum for pre-heating. Subsequently, the compression pressure was gradually increased from 0 MPa to 40 MPa in 10 min, and was maintained at 40 MPa for 5 min.

2.3. Characterization of nanocomposites

2.3.1. Tensile testing

Tensile properties were studied using a VGS1E strograph tensile test machine (Toyo Seiki Seisaku-sho, Ltd., Japan) equipped with a 50 N capacity load cell. The ASTM D638 standard dumbbell specimens were cut from the hot pressed sheets by ASTM Type 7 dumbbell shaped die cutter. The crosshead speed for the tensile measurement was 50 mm/min . The maximum load and elongation at break were recorded.

2.3.2. Nanoindentation measurements

The nanoindentation experiments were performed using a Nano Indenter G200 (TOYO Corporation, Japan). The normal load and loading speed for all the samples were set at $400 \mu\text{N}$ and $30 \mu\text{N s}^{-1}$, respectively. A Berkovich diamond indenter was used for performing the indentation experiments using a typical loading-hold-unloading sequence. After engaging the tip onto the sample surface, the load was steadily increased until the predefined maximum load was achieved. Then, in order to reduce the creep effect [17], the maximum load was retained for 10 s. Eventually, in the unloading cycle, the tip was withdrawn from the sample surface at the same rate. For each sample, at least five indents were recorded at different locations on the surface.

The hardness can be measured by analyzing the unloading part of the load-displacement curve. The indentation hardness is given by:

$$H = \frac{P_{\max}}{A} \quad (1)$$

where, P_{\max} is the maximum normal load and A is the projected contact area at the maximum load.

2.3.3. Friction and wear studies

A pin-on-disc friction (EFM-III-F Orientec Corporation, Japan) testing was conducted to measure the friction and wear resistance. Specimens with $1 \text{ cm} \times 1 \text{ cm}$ dimensions were cut from the hot-pressed sheets. The surface contact pressure, rotation speed, and testing time were set at 24 MPa, 100 rpm, and 10 min, respectively.

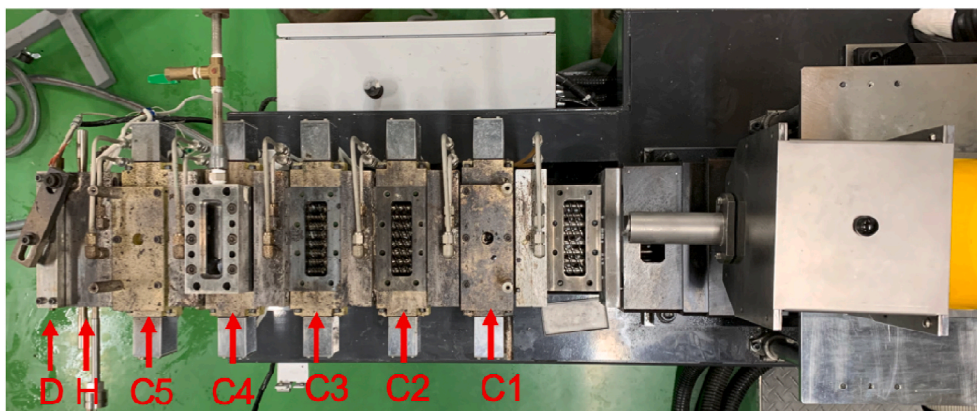


Fig. 1. Schematic of the octa-screw extrusion setup.

An optical surface profilometry studies (Zygo 3D, Tokyo, Japan) was conducted to observe the depth of specimens before and after the ring-on-disc friction and wear resistance tests. Scanning electron microscopy (JSM-6510, JEOL Ltd., Japan) studies were conducted to observe the wearing tracks subsequent to the friction and wear resistance tests under low vacuum conditions.

2.3.4. Differential scanning calorimetry (DSC)

Thermal property studies were carried out using differential scanning calorimetry (TA Instruments TA-Q200C, USA), at a heating rate of 3 °C/min between 40 and 400 °C. UHMWPE/graphene nanocomposites of approximately 5 mg were used in the measurement. The degree of crystallinity (X_c) was calculated according to the expression:

$$X_c(\%) = \left(\frac{\Delta H}{\Delta H_{100}} \right) \times 100 \quad (2)$$

where ΔH is the heat of fusion and ΔH_{100} (293 J/g) is the melting enthalpy of polyethylene with 100% crystallinity.

2.3.5. Thermogravimetric analysis (TGA)

TGA studies (TA Instruments Model QA-50, New Castle, DE, USA) were completed using about 10 (± 0.1) mg of UHMWPE/graphene nanocomposites which was heated from 40 °C to 500 °C. The heating rate was 3 °C/min and the test was operated with an air flow of 60 mL/min.

2.3.6. Transmission electron microscopy (TEM) studies

Transmission electron microscopy (JEM-2100F: JEOL Ltd.) studies with an acceleration voltage of 200 kV was conducted to examine the morphology of UHMWPE/graphene nanocomposites. The nanocomposite specimens were stained with ruthenium tetroxide (RuO_4) and then sliced into ultrathin sections in the order of 100 nm thickness by Ultramicrotome (Ultracut-UCT: Leica Microsystems).

2.3.7. Water contact angle (WCA) measurement

A water contact angle measuring instrument (Model DM 500, Kyowa Interface Science Co., Ltd.) was used to estimate the water contact angle of UHMWPE/graphene nanocomposites. Specimens with a dimension of 1 cm \times 1 cm were cut from the hot-pressed composite sheets. The specimen surfaces were carefully sprinkled with distilled water and were mounted on the testing plate. The contact angles were monitored and recorded using a video monitor.

2.3.8. Cell culture

Cytotoxicity of the printed meshes was examined by MTT assay (Roche, Germany) of cell viability. Graphene filled UHMWPE nanocomposites (60 nm, 1.5 wt%) were cut out with punch and put onto 24-well culture plates. Human fibroblasts were seeded (5×10^3 cells/well) in Dulbecco's Modified Eagle's Medium (DMEM) at 37 °C under 5% $\text{CO}_2/95\%$ air condition until cell confluence. Cell viability was monitored at 1–7 days by MTT assays and quantified using an ELISA reader.

3. Results and discussion

3.1. Mechanical strength

Stress-strain curves of graphene filled UHMWPE nanocomposites obtained from the tensile testing studies are shown in Fig. 2. It is noticed that the elongation at break has increased by 20.8% (from 10.1 to 12.2) with the addition of 1 wt% 60 nm graphene as compared to pure UHMWPE. This may be attributed to the increase in crystallinity with the presence of graphene in the nanocomposites as discussed in detail in section 3.3. However, with further increase in the graphene content, the mechanical property deteriorated. This behavior is mainly attributed to the lower percentage of the polymeric matrix that provides the base for

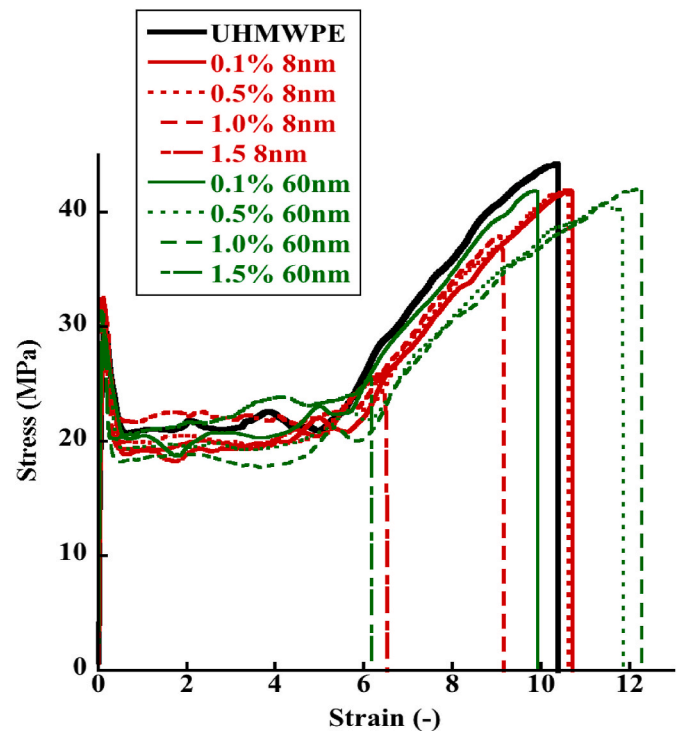


Fig. 2. Stress-strain curves of graphene filled UHMWPE nanocomposites.

strength. Table 1 presents the effect of graphene on the yield strengths of UHMWPE nanocomposites. The yield strength of UHMWPE/graphene nanocomposites shows improvement with the addition of graphene. Among the different variants of graphene used, the 8 nm graphene filled nanocomposites showed significant increase of yield strengths than the 60 nm graphene filled composites. Furthermore, the yield strength increased by 18% (from 28.5 MPa to 34.2 MPa) with the addition of 1.5% 8 nm graphene.

Nanoindentation is a powerful and advanced way of measuring the mechanical properties of various types of materials, and has been used widely to investigate the mechanical properties of polymers and polymeric composites. Fig. 3 shows the load-displacement curves of graphene filled nanocomposites, while the calculated hardness of the nanocomposites is listed in Table 1. Clearly, the addition of graphene

Table 1

Mechanical properties of graphene filled composites with different compositions of graphene (\pm the standard deviation).

Material	Filler content, %	Young's modulus, MPa	Yield strength, MPa	Elongation, %	Hardness (MPa)
UHMWPE	–	242 \pm 7	28.5 \pm 0.8	1010 \pm 87	480 \pm 2
UHMWPE/8 nm	0.1	263 \pm 5	29.6 \pm 1.0	1070 \pm 14	530 \pm 1
	0.5	278 \pm 4	30.6 \pm 0.4	1050 \pm 45	590 \pm 1
	1.0	329 \pm 6	32.11 \pm 1.3	920 \pm 62	630 \pm 1
	1.5	351 \pm 8	34.2 \pm 0.7	650 \pm 33	690 \pm 3
UHMWPE/60 nm	0.1	257 \pm 4	29.4 \pm 0.3	980 \pm 44	510 \pm 0
	0.5	269 \pm 11	29.8 \pm 0.9	1180 \pm 72	550 \pm 3
	1.0	305 \pm 4	30.4 \pm 0.6	1220 \pm 14	600 \pm 0
	1.5	341 \pm 8	31.0 \pm 0.9	620 \pm 24	660 \pm 3

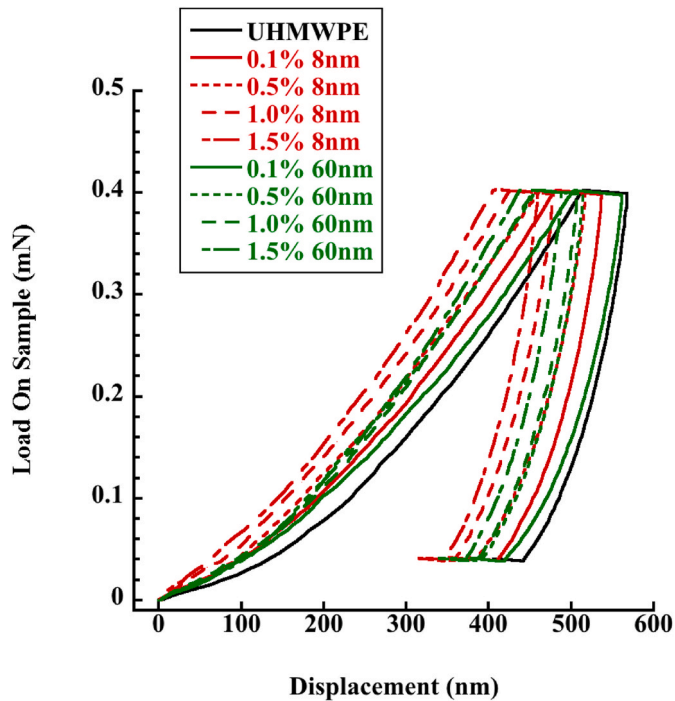


Fig. 3. Load-displacement curves of graphene filled UHMWPE nanocomposites for the nanoindentation tests.

has increased the hardness of the composites. The existence of graphene nanofillers present some obstacles in the matrix [17]; as a result, the indenter needs more force to overcome these obstacles. The hardness increases accordingly.

3.2. Wear resistance

It has been reported that the wear debris due to the in vivo friction of artificial joint may lead to osteolysis, bone loss, artificial joint loosening, and decrease the lifespan of artificial joint significantly [14]. It is thus important to examine the wear resistance of nanofiller reinforced UHMWPE. This work employed the pin-on-disc method to evaluate the wear resistance of graphene filled UHMWPE nanocomposites. Pin-on-disc measurement has been widely employed to assess the wear rate (by weight or by volume) [18–20]. The wear resistance of virgin UHMWPE and graphene filled UHMWPE composites are shown in Fig. 4. In the case of the 8 nm graphene filled UHMWPE, the wear resistance increases when the graphene content is low, at 0.5 and 1.0 wt%. However, as the content of graphene is increased to 1.5%, the wear resistance decreases significantly. On the other hand, 60 nm graphene filled composites have shown inferior wear resistance compared to pure UHMWPE. Fig. 5 shows the post wear test SEM images of virgin UHMWPE, 0.5% 8 nm graphene filled UHMWPE and 0.5% 60 nm graphene filled UHMWPE. The surface roughness of the 60 nm graphene filled composites is noticed to be much higher than those of pure UHMWPE and 8 nm graphene filled composites. Due to their smaller size, the 8 nm graphene fillers provide superior mechanical locking at the filler/matrix interface. In this regard, UHMWPE composites show greater wear resistance than pure UHMWPE. In comparison with the smaller sized 8 nm graphene, the 60 nm graphene may possess less mechanical locking capability and are easily ejected from the filler/matrix interfaces. Such fillers act as grinding particles at the wear interface which contributes for the increase in wear and reduction of wear resistance.

Tai et al. [9] have reported that the wear resistance of UHMWPE/graphene nanocomposites decreases up to 0.7 wt% addition of graphene and shows no significant influence on wear rate for higher

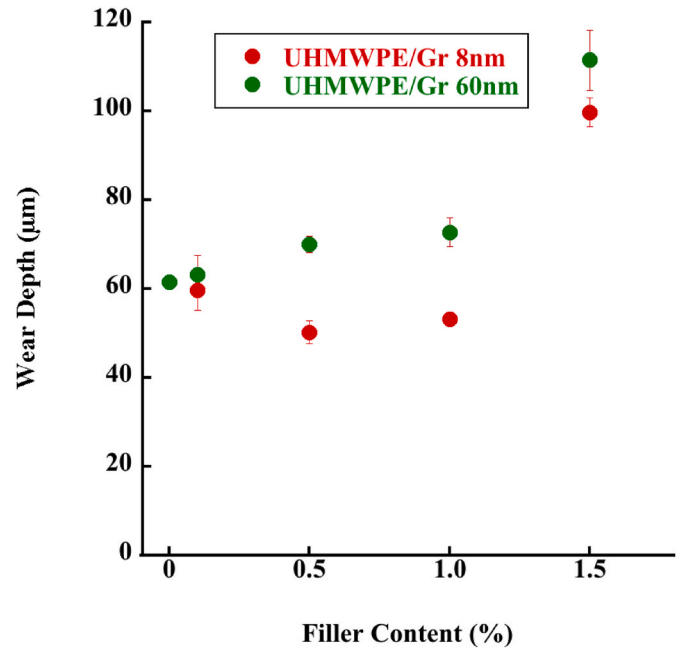


Fig. 4. Wear depths of UHMWPE nanocomposites with different content of graphene.

concentrations. The current experimental results show that the wear resistance increases with the content of 8 nm graphene in the nanocomposites up to 1.0 wt%. With the excellent compounding capability of octa-screw extruder, UHMWPE nanocomposites with a higher filling content of graphene and the relevant wear resistance property has been achieved. The experimental data in this study has shown an increase of wear resistance with the addition of graphene. The results indicate that the addition of graphene to UHMWPE matrix might be a potential solution to reduce the negative influence of irradiation/oxidation on the long-term properties of UHMWPE implants. Panin et al. [21] investigated the influence of material modification by nano-hydroxyapatite (nHA) particles on tribotechnical properties of UHMWPE using a “block-on-ring” scheme, and found that the addition of 0.1–0.5 wt% nHA leads to an increase of wear resistance by 3 times (despite of a reduction of the ultimate tensile strengths). It has been reported that the block-on-ping method generally shows lower wear rate and low friction coefficient compared to pin-on-disc scheme [22] used in this study. More experimental works may thus be needed in the future to compare the wear resist of nHA and graphene filled nanocomposites by employing the test method. Additionally, graphene-based HA nanocomposites may also be exploited to further promote the wear resist properties for potential clinical applications.

3.3. Thermal properties

The variation in the melting temperature of UHMWPE composites due to the addition of graphene is examined as shown in Fig. 6. The results suggest that the melting temperature is unaffected by the addition of both 8 nm and 60 nm graphene nanofillers.

The thermal analysis of all the samples in Fig. 7 show that the crystallinity has increased from 56% to 67%. The experimental data indicate that the addition of graphene with concentration less than 1.5 wt%, has limited influence on both the melting temperature and crystallization of the nanocomposites. More profound influence of graphene on the degree of crystallinity of the nanocomposites have been reported. Todd et al. [23] have reported reduced crystallinity of up to 9% for a thermally reduced graphite oxide reinforced polyethylene composite as compared to virgin polyethylene. Martinez-Morlanes et al. [24] found lower crystallinity for multi-walled carbon nanotube filled UHMWPE.

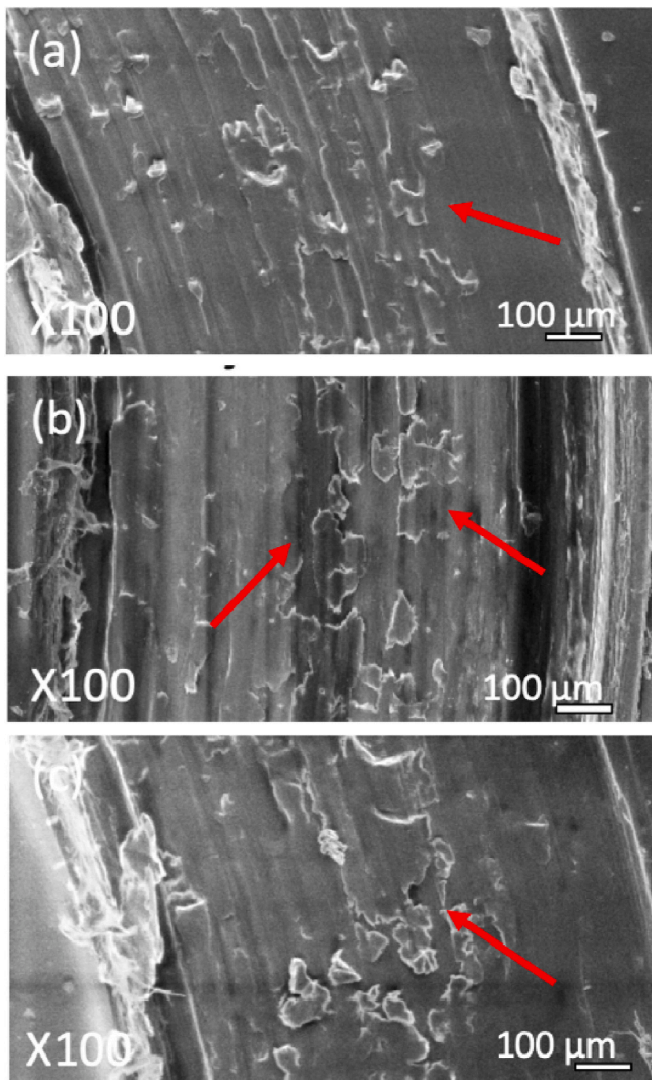


Fig. 5. SEM images of nanocomposites after wear test, (a) virgin UHMWPE (b) UHMWPE/8 nm graphene, (c) UHMWPE/60 nm graphene.

Nevertheless, a higher degree of crystallinity introduced by graphene addition has also been proposed [11]. These discrepancies may result from the characteristic of the nanofillers and their capability in achieving bonding at the filler/polyethylene interfaces.

3.4. Thermogravimetric analysis

The variation of decomposition temperature of virgin UHMWPE and graphene filled UHMWPE nanocomposites is compared in Fig. 8. The decomposition temperature of nanocomposites increase with the graphene content. With the addition of 1.5 wt% 60 nm graphene, the decomposition temperature of UHMWPE increases significantly from 412 °C to 430 °C.

The TGA studies confirm improved thermal stability of UHMWPE with the addition of graphene. Before implantation, UHMWPE implants conventionally receive irradiation treatment and post-irradiation processing/storage, thus leading to natural aging [25,26] and oxidative degradation. Suner et al. [27] have reported that the addition of graphene oxide into UHMWPE decreases the oxidation degradation induced fracture stress reduction. Sreekanth et al. [28] and Martinez-Morlanes et al. [24] have identified the positive influence of multi-walled carbon nanotubes in promoting the oxidative stability of UHMWPE. The experimental results in this study suggest that graphene possesses the

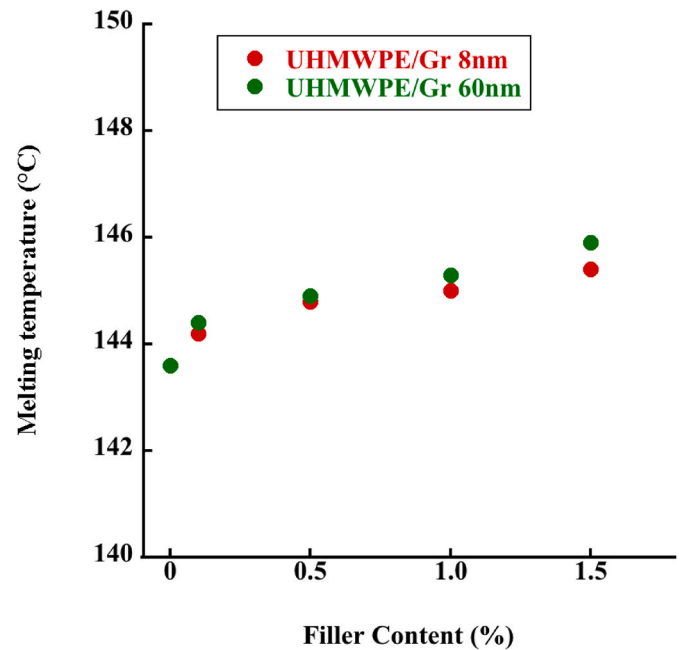


Fig. 6. Melting temperature of graphene filled composites with different concentration of graphene.

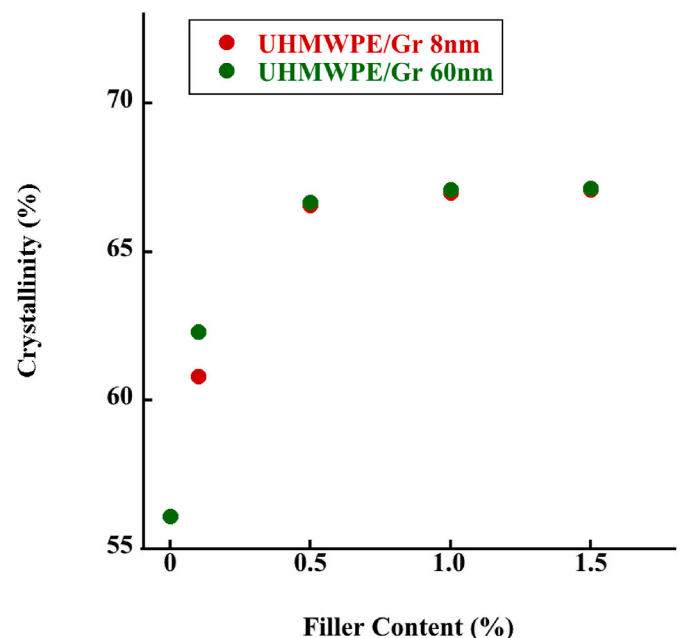


Fig. 7. Crystallinity of graphene filled composites with different graphene content.

ability to relieve the influence of free radicals during the thermal decomposition process.

3.5. Internal morphology

TEM studies were conducted to assess the dispersion properties of UHMWPE nanocomposite reinforced with the two grades of graphene (namely, 8 nm and 60 nm). Fig. 9(a) and (b) show uniform dispersion and absence of aggregation of graphene filler in the polymer matrix, illustrating the excellent compounding ability of the octa-screw extruder. In addition, the growth of lamellae originating from

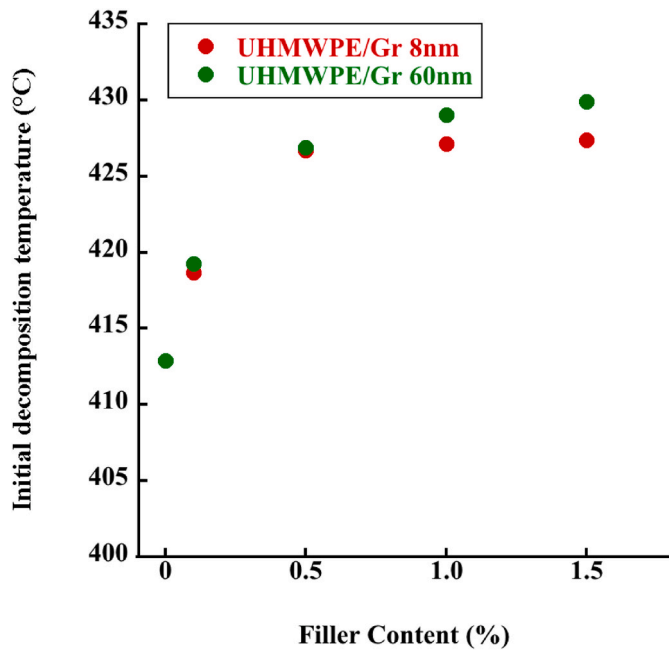


Fig. 8. Decomposition temperature of graphene filled composites with varying graphene concentration.

graphene in the matrix is also observed from the TEM images. This suggests that the addition and dispersion of graphene has influence on the crystallinity of UHMWPE.

3.6. Water contact angle

Fig. 10 shows that the water contact angles were 93.4, 91.1, 89.4, 86.5, and 85.2° for the pure UHMWPE, and 0.1%, 0.5%, 1.0% and 1.5% graphene added nanocomposites, respectively. Clearly, the water contact angle is closely related to the content of graphene and improves the hydrophilicity of nanocomposite. Increasing the friction between the bearings may result in improving the lubrication of the joints [29] which

is a concern in virgin UHMWPE. In this regard, various researchers have endeavored to enhance the hydrophilicity by modifying the surface of polyethylene or by adding nanoparticles [30,31]. The results have demonstrated that the addition of graphene promotes the wettability of UHMWPE, enhances lubrication, reduces friction and improves the tribological performance of artificial joints [32].

3.7. Cytotoxicity of nanocomposites

For medical applications such as implants, good biocompatibility of the materials is a necessity. Fig. 11 shows the measured cell viability at 1–7 days. Graphene filled UHMWPE nanocomposites show no signs of cytotoxicity. This further demonstrates the ability of graphene to promote the performance of UHMWPE and the potential applications of UHMWPE nanocomposites for joint replacements.

4. Conclusions

This work has studied the effect of graphene addition on the performance of UHMWPE. UHMWPE/graphene nanocomposites with varied concentrations of graphene are satisfactorily compounded employing octa-screw extrusion process. Microscopy studies suggest the absence of aggregation and uniform dispersion of graphene filler, confirming the excellent compounding ability of the octa-screw extruder. The result of thermal analysis shows that the crystallinity increases from 56% to 67% with the addition of graphene. With the addition of 1.5 wt% 60 nm graphene, the decomposition temperature of UHMWPE increases by 4% from 412 °C to 430 °C. The yield strength increases by 18% (from 28.9 MPa to 34.2 MPa) with the addition of 1.5% 8 nm graphene, while the elongation at break increases by 20.8% (from 10.1 to 12.2) with the addition of 0.5 wt% 60 nm graphene. Incorporation of 0.5% and 1.0% 8 nm graphene filled composites showed superior wear resistance as compared to pure UHMWPE. Hardness and the hydrophilicity of the nanocomposites increases with inclusion of graphene fillers in the composites. Graphene filled UHMWPE nanocomposites showed no signs of cytotoxicity. Thus the UHMWPE/graphene nanocomposites prepared by octa-screw extrusion exhibit superior potential for prospective joint replacement applications.

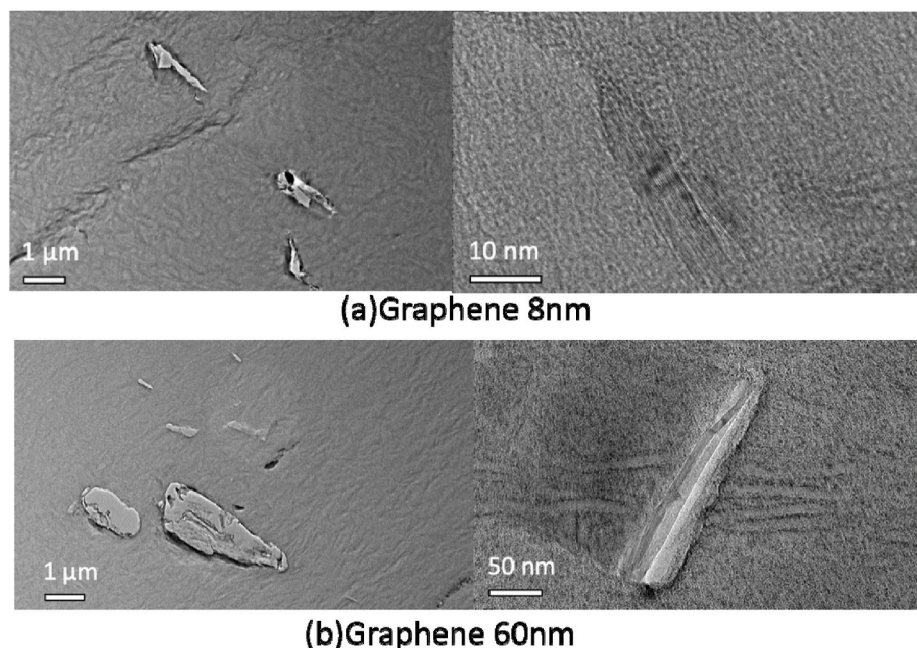


Fig. 9. TEM images of (a) 8 nm, (b) 60 nm graphene filled nanocomposites.

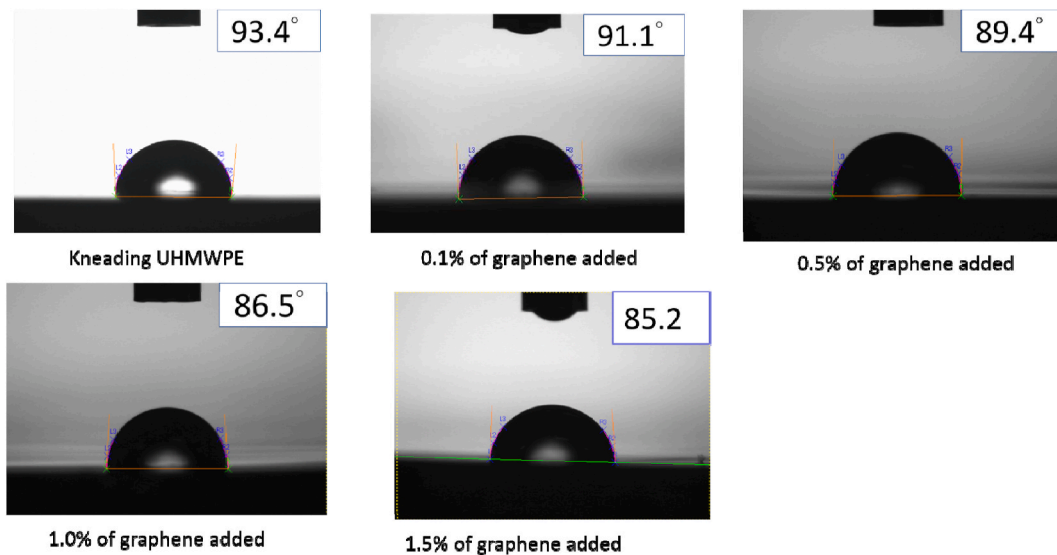


Fig. 10. Water contact angles of graphene incorporated nanocomposites.

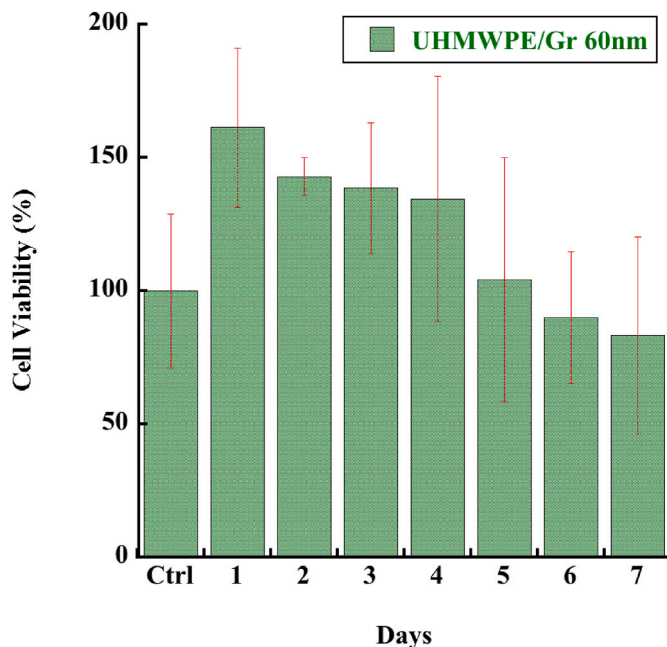


Fig. 11. Cytotoxicity of graphene filled nanocomposites.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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