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ENHANCEMENT OF MECHANICAL PROPERTIES OF UHMWPE POLYMER BY NITROGEN ION IMPLANTATION

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ABSTRACT

The use of ion implantation as a surface treatment technology has been conquered by its applications to prevent wear and oxidation in metal alloys, even though some early works already pointed out that ion implantation could also be effective as a surface treatment for other materials, including polymers. Further research has shown that low dose implantation of energetic light ions could be very effective for improving properties such as wear resistance and hardness in many different polymers. Cross linking of polymeric chains due to ionization energy provided by the stopping process is the main mechanism to explain the changes in mechanical properties. According to this model the lighter the ion is, the stronger is the effect. This thesis presents the results obtained by nitrogen implantation in ultra-high molecular weight polyethylene (UHMWPE). N+ ions were implanted at 80 keV, 100 keV and 120 keV energy levels which are maintained at a fluencies or dose of 5×10^{15} ions/cm^2. Wear resistance was measured using pin-on-disc wear testing machine at a constant load of 18 kg or 177N with a sliding speed of 2 m/s for about 1000m sliding distance. The results clearly show a lower weight and volume loses for samples implanted with nitrogen, in comparison to those implanted with untreated samples. Hence from the results of weight and volume loses of the treated or implanted samples with nitrogen ion, gives very good wear resistance than untreated samples.

Keywords: *UHMWPE, Nitrogen ion, polymers, Ionization energy, Ion implantation, Polyethylene and wear resistance.*

1. Introduction

1.1 Bio-Engineering

Bioengineering is an application of engineering principles and design to challenges in human health and medicine. Bioengineering is related to Biological Engineering, the latter including applications of engineering principles to the full spectrum of living systems, from microbes and plants to ecosystems. Bioengineering exploits new developments in molecular biology, biochemistry, microbiology and neurosciences as well as sensing, electronics and imaging, and applies them to the design of medical devices, diagnostic equipment, biocompatible materials, and other important medical needs.

Wilches et al. [40] reviewed that Bioengineering couples engineering expertise with knowledge in biological sciences such as genetics, molecular biology, protein chemistry, cytology, neurobiology, immunology, physiology and pharmacology. Bioengineers work closely with but are not limited to, medical doctors and other health professionals to develop technical solutions to current and emerging health concerns.

1.2 Bio-Problems

Illingworth et al. [49] stated that wear of the artificial implant material and resulting wear debris induced osteolysis is a major cause of failure in both hip and knee prosthesis. Ultra-high molecular weight polyethylene (UHMWPE) wear particles generated at the articulating interfaces enters the periprosthetic tissues, where they are phagocytosed by macrophages which release inflammatory cytokines such as TNF alpha and lead to bone resorption. Over the last forty years, different types of UHMWPE have been used clinically. Historically UHMWPE sterilized with gamma irradiation in air was used, and this was subsequently shown to oxidize and degrade with age resulting in accelerated wear. More recently stabilized UHMWPE and cross-linked UHMWPE have been introduced.

The wear, wear debris, biological activity and osteolysis potential of different types of UHMWPE have been investigated in laboratory simulations, under a range of different conditions. Laboratory studies of

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historical materials have been compared to clinical retrievals. The importance of investigating the nature of the wear debris and its biological activity as well as the wear rate has been demonstrated, with various types of UHMWPE debris showing different levels of biological activity. Integration of the results of wear simulator studies and in vitro cell culture studies has allowed the prediction of the relative osteolytic potential of different UHMWPE materials. The technology of high intensity nitrogen ion beams was perfected in the course of nuclear fusion programs. Since the development of the nitrogen ion implantation process by Harwell, numerous companies have tried to market the technique for surface modification of metals.

Federal et al. [44] described that a typical direct nitrogen ion implanter nitrogen gas is fed into the ion source, where electrons emitted from a hot filament to ionize the nitrogen atoms and molecules, forming a plasma. Nitrogen ions are then accelerated through a voltage drop, typically about 100 keV. The accelerated beam of nitrogen ions is directed at the surface of the 22 part to be implanted in the vacuum chamber. Budzynski et al. [41] studies related to Nitrogen ion implantation show increases in wear and fatigue resistance, lubricity, and in some cases, corrosion resistance of metal surfaces. In addition, nitrogen ion implantation has been found to increase the wear life of parts treated with hard chromium electroplate by between 5 and 10 times. Nitrogen ion implantation has the added environmental benefit of reducing the need to perform the hard chromium electroplating process by extending the life of the electroplated coating (2006, Ferber et. al. 1991, Fischer et. al. 1991).

Allen et. al. [48] have studied 'N' ion implantation on polymers and again found that wear resistance was improved and Kim beck RT et. al. [49] reported that improvement in surface wettability after implantation.

1.3 Problem Definition

The professional practice of engineering is largely concerned with design is an important aspect which play a vital role. A poor design may cause heavy loss to society in usage and economically many aspects play a major role which if not considered much many leads to failure. Many parameters influence the failure such as consideration of property of the material, erection, design, manufacturability and treatments.

But, in the field of biomechanical, the situation turns crucial once the component fails. Though they are capable to satisfy the bio-functionality, interfacing surface in relative motion causes friction, wear causes damage to the one or both surfaces generally involving progressive loss of material. Worn surface will produce wear debris which influences the host failing its bio-functionality. As the manufacturing engineer cannot eliminate the wear completely so we can study the material characteristics by which manufacturer will be befitted. In this report the material Ultra high molecular weight polyethylene is studied. Characterization is to be done to the material by studying its properties.

1.4 Bio-Compatibility

In surgery, a biocompatible material (sometimes shortened to biomaterial) is a synthetic or natural material used to replace part of a living system or to function in intimate contact with living tissue. Biocompatible materials are intended to interface with biological systems to evaluate, treat, augment or replace any tissue, organ or function of the body. Biomaterials are usually nonviable, but may also be viable. A biocompatible material is different from a biological material such as bone that is produced by a biological system. Artificial hips, vascular stents, artificial pacemakers and catheters are all made from different biomaterials and comprise different medical devices.

1.5 Knee joint

The knee is the lower extremity joint connecting the femur, fibula, patella, and the tibia. Since in humans the knee supports nearly the entire weight of the body, it is the joint most vulnerable both to acute injury and the development of osteoarthritis. Upon birth, a baby will not have a conventional knee cap, but a growth formed of cartilage. In human females this turns to a normal bone knee cap by the age of 3, in males the age of 5.

2. Ultra-High Molecular Weight Polyethylene (UHMWPE)

2.1 Introduction to UHMWPE

Ultra-High Molecular Weight Polyethylene (UHMWPE) is an engineering polymer that varies from high-density polyethylene (HDPE) in terms of average molecular weight and average chain length [1]. According to the International Standards Organization (ISO), UHMWPE has a molecular weight of at least 1 million g/mole and degree of polymerization of 36,000, while according to the American Society for Testing and Materials (ASTM) it has a molecular weight of greater than 3.1 million g/mole and degree of polymerization of 110,000 [2]. The properties of UHMWPE are highly dependent on their microstructure rather than molecular mass [3]. UHMWPE is a semi-crystalline polymer that contains fully crystalline and fully amorphous phases as an interfacial all-trans phase [4,5].

In the crystalline phase, the particular lamellar shape of crystallite is due to the chain folding with the chain axis, which enlarges the chain fold area. In the amorphous phase, the chains are interconnected through occasional cross links and random entanglements instead of proper chain folding. The relation between amorphous and crystalline phases is provided by tie molecules. The crystallinity of UHMWPE depends on its volumetric percentage of crystallites [6]. The properties of UHMWPE are determined by the connections between amorphous and crystalline phases, i.e., tie molecules, crystallinity, the degree of cross links and entanglements, and the positions of the crystallites. The average properties of UHMWPE and HDPE are presented in Table 1. Fig. 1 shows the structure of UHMWPE.

Table 1 Average properties of Ultra-High Molecular Weight Polyethylene (UHMWPE) and high-density polyethylene (HDPE) [7]

Property	UHMWPE	HDPE
Melting temperature (◦C)	132–138	130–137
Molecular weight (106 g/mol)	3.5–7.5	0.05–0.25
Specific gravity	0.925–0.945	0.952–0.965
Poisson's ratio	0.46	0.40
Modulus of elasticity (GPa)	0.5–0.8	0.4–4.0
Tensile ultimate strength (MPa)	39–48	22–31
Tensile yield strength (MPa)	21–28	26–33
Tensile ultimate elongation (%)	350–525	10–1200
Degree of crystallinity (%)	39–75	60–80
Impact strength (J/m of notch)	1070	21–214
Wear Rate (mm3 /106 cycles)	80–100	380–400

UHMWPE has high wear-resistance, toughness, durability, and biocompatibility. Therefore, it is commonly used as a bearing material with ceramic or metallic counter surfaces in joint arthroplasty [8,9] UHMWPE's significance for achieving outstanding performance in total joint arthroplasties is unquestionable [10,11]. For long-term clinical applications, its tribological performance and lifetime are key aspects [12,13]. However, UHMWPE implants have limited life due to their wear complications. When the UHMWPE is used in the periprosthetic environment it induces osteolysis followed by loosening of the implant. This implant loosening is joined with fatigue causes the aseptic loosening which ultimately causes the implant's failure. [14–17]. Many methods such as improving cross-linking [18–21], or crystallinity percentage [22–25] through irradiation [26], surface modification through plasma treatment [27,28], or introducing effective textures [29,30], and reinforcements with particles or fibers [31–33] have been used for enhancing properties of UHMWPE. Few review articles [2, 18, 34] have been published to correlate the mechanics and morphology of UHMWPE with its wear and mechanical properties. In one review [35], the influence of CNT and graphene as reinforcements for UHMWPE is evaluated.

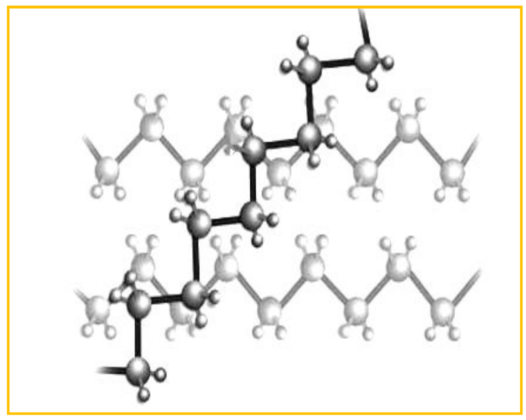

Fig. 1 Structure of UHMWPE

In a few review articles [3, 36], other advances in UHMWPE for improving wear and mechanical performance are discussed. However, in such articles, many studies on other polymeric materials are considered for supporting the evidence and there is a lack of clarity regarding the optimal values of the effective methods. The objective of this study is to summarize the existing practices for the enhanced tribological and mechanical performance of UHMWPE. The influence of irradiation, reinforcements and surface modifications is briefly discussed and a tabular data is presented for estimating the optimal values or materials. As a conclusion, by using the UHMWPE, mechanical and tribological findings were further improved in order to provide durable implants in patients.

3. Nitrogen Ion Implantation

3.1 Introduction

Nitrogen Ion Implantation for surface modification of metals was pioneered in the United Kingdom (UK) by Harwell Laboratory in the early 1970s. Nitrogen was chosen because the intense nitrogen beams are easily produced.

Ion implantation techniques are increasingly being employed as a final treatment to modify the surface characteristics of technological polymers. Tribological or Bio-compatibility, mechanical properties can be improved by high energy, low-dose ion bombardment. Some previous works were carried out to increase polymer wear resistance by using low-dose nitrogen implantation. Some relevant changes have been detected, as well as a darkening of the material that does not allow further optical uses.

Fundamental studies have explained how the cross linking of polymeric chains due to ionization energy provided by the stopping process becomes the mail mechanism to explain the observed improvements in mechanical properties. According to this model the lighter the ion is the stronger is the effect. Low-dose implantation of energetic light ions can be very effective treatment to improve properties such as wear resistance and hardness in many different polymers, such as the UHMWPE employed in hip, knee and elbow prostheses. Nitrogen produce positive effects and it is easier to implant using commonly available industrial implanters. The objective of this work is to evaluate the effect of light nitrogen ions implanted in UHMWPE samples.

3.2 Experimental details

UHMWPE used has an average molecular weight of 5,000,000 g/mol and density of 0.945 g/cm^3. UHMWPE was implanted by 80 keV, N^+ ions with the dose $5x10^{16}$ ions/cm^2 by 150keV ion accelerator. During implantation, the pressure of the chamber was $3x10^{-7}$ mbar. The beam current density was less than 0.1 mA/cm^2. It was found that all the polymeric materials will melt at higher temperatures and as far as UHMWPE is concerned its melting point is around 150^0 C. Hence the ion implantation process for this material is carried at room temperature. Fig. 2 shows the setup of 150 keV ion implanter.

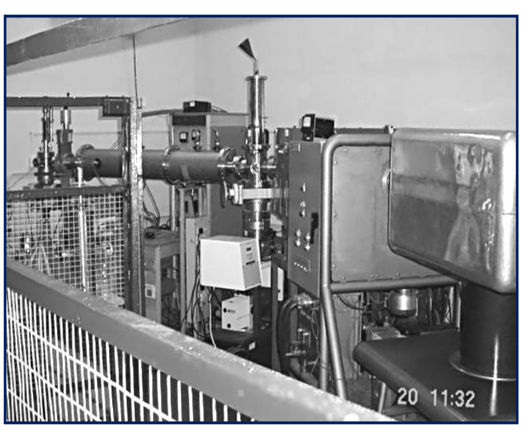

Fig. 2 Setup of 150 keV ion implanter

The UHMWPE samples were implanted with N^+ ions at 80 keV, 100keV and 120 keV energy levels using the ion implanter at a base pressure. The nitrogen ions are produced from the nitrogen gas, the gas enters the ion source where it is converted into nitrogen ions with the help of R F Oscillator and the ions that are formed gets energized due to the high voltage power supply connected to ion source. The energized ions enter into the accelerating tube where these ions gain the acceleration and enter the mass analyzer magnet. Here the magnets inside makes the ions to travel towards the target chamber where UHMWPE material is placed. As the material is insulator, we made it as a conductive with the help of metallic paint. The ions are bombarded onto the material until the dose is reached to $5*10^{16}$ ions/cm^2.

4. Wear Testing

4.1 Pin on Disc Tester

A pin on disc tester is the standard equipment used to determine the sliding friction coefficient and wear resistance of surfaces. The tester consists of a stationary "pin" under an applied load in contact with a rotating disc. Either the pin or the disc can be wear- and friction-tested using the pin on disc tester. The pin is usually a sphere however it may be any geometry that simulates the actual application counter surface. A load cell attached to the pin on disc tester is used to measure the evolution of the friction coefficient with sliding distance. Sliding wear of the disc can be measured after the pin on disc test using a simple piece of equipment called a Calo tester. The pin on disc test has proved particularly useful in providing a simple wear and friction test for low friction coatings on machine components, such as the valve train, particularly in motor sports. These components are now coated with low friction coatings such as diamond like carbon to reduce energy losses and the requirement for lubricant.

4.2 Experimental Details

Dry sliding wear tests were conducted on a pin-on-disc friction and wear testing machine. The cylindrical pin specimens of size 10 mm diameter and 16mm length were tested against SS316L (Stainless steel 316L grade) disc. The tests were conducted at a load of 18kg or 177N and with sliding velocity of 2 m/s. the weight loss measurement was taken after running the test for 1000m of sliding distance. Although using 2 m/s sliding speed led to test duration of 8.33 min (8 min 20 sec), it was insured that the test was carried within the steady-state region.

The wear rates were calculated from the weight loss measurements. Fig. 3 shows the schematic diagram of the pin-on-disc wear test rig used for this work. The rig consists of a stainless-steel table which is mounted on a turntable, a variable speed motor which provides the unidirectional motion to the turntable and hence to the disc sample and a pin sample holder which is rigidly attached to a pivoted loading arm. This loading arm is supported in bearing arrangements to allow loads to be applied to the specimen. During the test, friction force was measured by a transducer mounted on the loading arm.

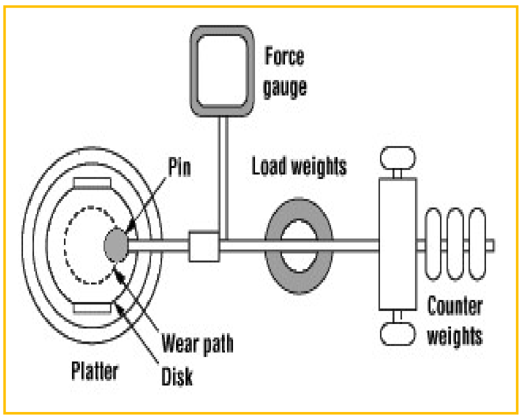

Fig. 3 Pin-On-Disc Wear Test Rig

Polymer materials used in this investigation and the specific test conditions like sliding velocity, sliding distance and load applied were given below

Sliding velocity = 2 m/s.
Sliding distance = 1000m.
Load applied = 18kg or 177N.

5. Equations and Symbols

The formulae used in calculating wear are:

Weight loss = Initial weight – Final weight, grams

Volume loss = weight loss/density,mm^3

Wear = volume loss / sliding distance, mm^3/m

Wear factor =volume loss/(sliding distance* load), mm^3/Nm

% Vol-loss l=[(vol loss of treatedsample)/(vol loss of untreated sample)] * 100

6. Results and Discussion

6.1 Weight-loss of samples due to wear

From Fig. 4, the untreated UHMWPE samples results in a weightloss due to wear ranges from 0.0037 grams to 0.0040 grams.

From Fig. 5, the weight loss of the treated sample with 80 keV energy level is 0.0005 grams which is less compared to 100 keV and 120 keV energy levels with weight loss of 0.0014 grams and 0.0027 grams respectively. Hence we can understand from this is that the treated sample with energy 80 keV had good wear resistance than 100 keV,120 keV and Untreated samples.

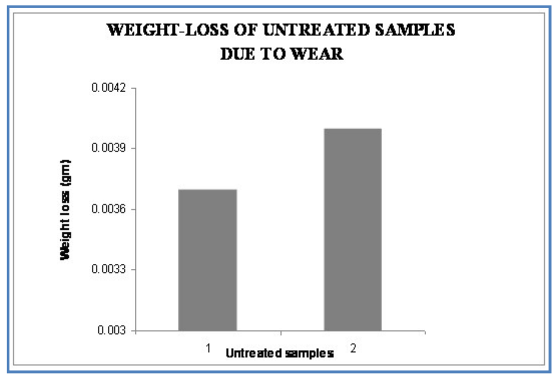

Fig. 4 Graph for weight-loss of untreated samples due to wear

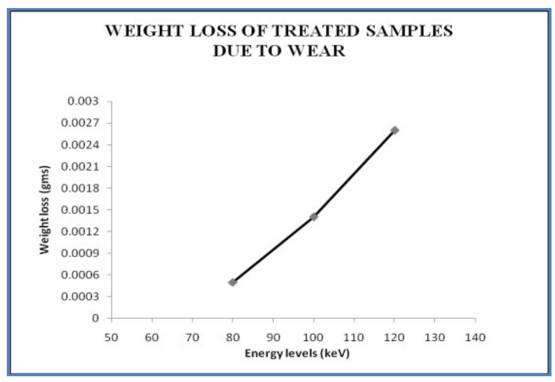

Fig. 5 Graph for weight-loss of treated samples due to wear

6.2 Volume-loss of untreated samples due to wear

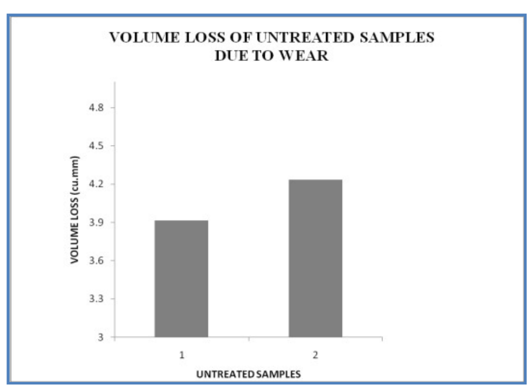

Fig. 6 Graph for volume-loss of untreated samples due to wear

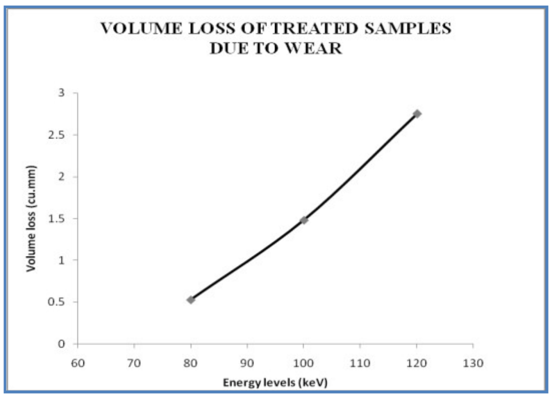

Fig. 7 Graph for volume-loss of untreated samples due to wear

From Fig. 6, the untreated UHMWPE samples results in a volumeloss due to wear ranges from 3.9153 mm^3 to 4.2328 mm^3.

From Fig. 7, the volumeloss of the treated sample with 80 keV energy level is 0.5291 mm^3 which is less compared to 100 keV and 120 keV energy levels with volumeloss of 1.4815 mm^3 and 2.7513 mm^3 respectively. Hence we can infer from this that the treated sample with energy 80 keV had good wear resistance than 100 keV, 120 keV and Untreated samples.

6.3 %Volume-loss of treated samples due to wear

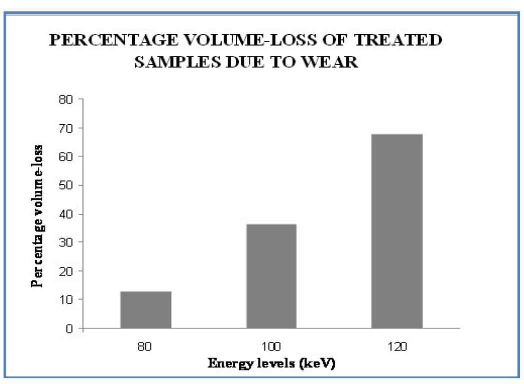

Fig. 8 Graph for % volume loss of treated samples due to wear.

Fig. 8 show the volume loss percentage treated samples due to wear. The percentage volume loss of the treated samples is low compared to the untreated samples and the treated sample with 80keV energy level had a volume loss of only 7.8% of volume loss of untreated sample which is treated as the best result apart from the three energy levels.

6.4 Scanning Electron Microscopy (SEM) Analysis

In order to investigate the effect of Nitrogen Ion Implantaion on the UHMWPE micro structure during the unidirectional sliding wear SEM analysis was carried out with a JEOL MODEL JSM-6360 SEM.The evolution of the effect of wear on the surface of the ion implanted i.e treated sample pin with 80keV energy level and untreated pin was examined for the comparision with sliding wear process. From Fig. 9 and Fig.10, we can see that micro shearing and micro cracks are evolved for the untreated samples when undergone to sliding wear. From Fig. 11 & Fig. 12, we can see that the treated samples with 80keV energy level SEM images which clearly says that there is a difference in the surface morphology as compared to the untreated samples. Here treated sample with 80keV energy level is prefered for comparing with untreated sample because it was already proved from the wear test results that it has the good wear resistance compared to the samples with energy levels 100keV and 120keV.

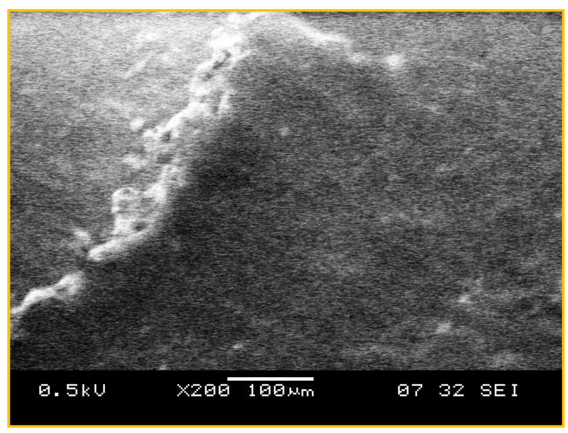

Fig. 9 Micro shearing flow

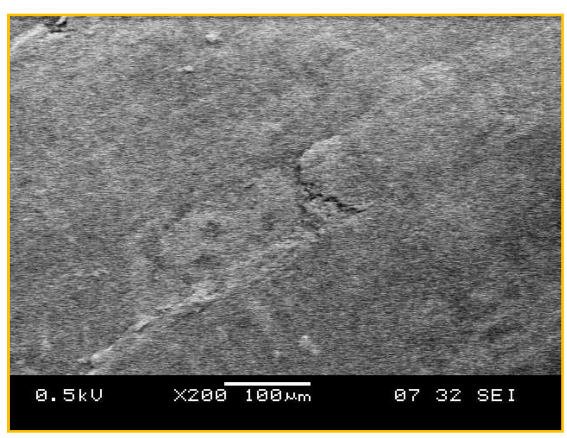

Fig. 10 Micro cracks.

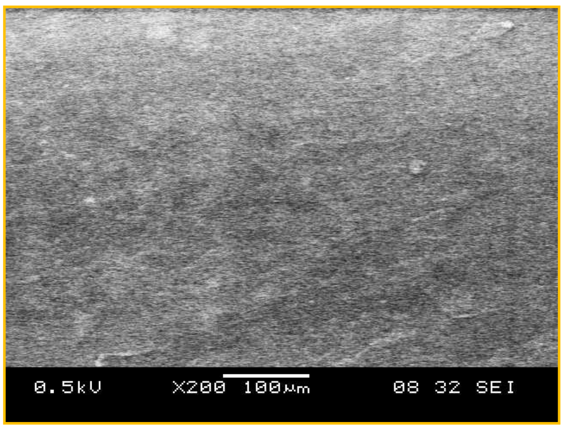

Fig. 11 Surface morphology of untreated sample

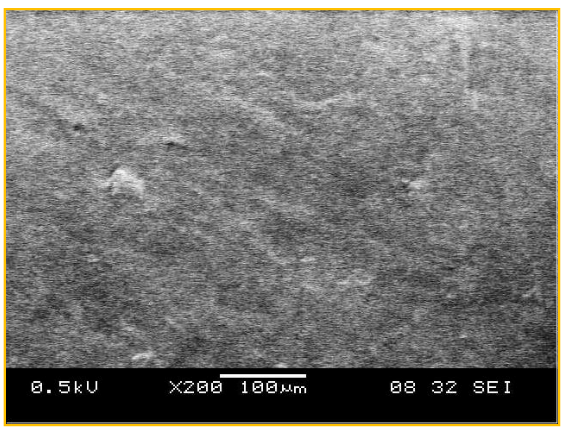

Fig. 12 Surface morphology of treated sample

7. Conclusion

- **i.** Obtained results shows that the polymeric material (UHMWPE) suffers modification when subjected to nitrogen ion implantation. Ion implantation smoothens the implanted surface area reducing the friction.
- **ii.** Good wear resistance was observed for the sample which was resulted with minimum weight and volume losses .
- **iii.** The weight loss of treated sample with energy level of 80 keV for a dose of $5x10^{16}$ ions/cm^2 is 0.0005 grams which is far better compared to 100 keV, 120 keV and Untreated samples with weight loss of 0.0014 grams, 0.0027 grams and (0.0037 grams – 0.0040 grams) respectively.
- **iv.** The volume loss of treated sample with energy level of 80 keV for a dose of $5*10^{16}$ ions/cm^2 is 0.5291 mm^3 which is far better compared to 100 keV, 120 keV and Untreated samples with volume loss of 1.4815 mm^3, 2.7513 mm^3 and (3.9153 mm^3 – 4.2328 mm^3) respectively.
- v. The percentage volume loss of the treated samples is low compared to the untreated samples and the treated sample with 80keV energy level had a volume loss of only 7.8% of volume loss of untreated sample which is treated as the best result apart from the three energy levels.
- vi. The treated samples with 80keV energy level sem images which clearly says that there is a difference in the surface morphology as compared to the untreated samples.

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