

# Surface characterization of ultra high molecular weight polyethylene modified by swift heavy ion beam bombardment

M.F. del Grosso<sup>a,\*</sup>, V.C. Chappa<sup>a,b</sup>, G. García Bermúdez<sup>a,b,c</sup>, E. Forlerer<sup>d</sup>, M. Behar<sup>e</sup>

<sup>a</sup> U. A. de Física, Tandara, CNEA, 1429 Buenos Aires, Argentina

<sup>b</sup> Consejo Nacional de Investigaciones Científicas y Técnicas, Argentina

<sup>c</sup> Escuela de Ciencia y Tecnología, UNSAM, 1653 Buenos Aires, Argentina

<sup>d</sup> U. A. Materiales, CAC, CNEA, (1429) Buenos Aires, Argentina

<sup>e</sup> Instituto de Física, UFRGS, Caixa Postal 15051, 91501-970, Porto Alegre, RS, Brazil

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## Abstract

The damage processes induced by swift heavy ions (SHI), can be very different to those induced by classical low ionising particles. This is due to the high electronic stopping power of SHI. Ultra high molecular weight polyethylene (UHMWPE) was irradiated with 6.77 MeV helium and 12.5 MeV carbon beams and fluences ranging from  $10^{11}$  to  $10^{13}$  cm<sup>-2</sup> and  $2 \times 10^{10}$  to  $5 \times 10^{13}$  cm<sup>-2</sup>, respectively. Structural changes at the polymer near surface region were studied by means of infrared spectroscopy measurements and wear resistance tests. With FTIR spectroscopy we studied the changes in crystallinity, double bond C=C, trans-vinylene and graphite formation and the evolution of methylene group as a function of fluence. The experiments have determined that exists an optimum ion fluence value, that depends on the ion mass and energy, at which the wear resistance increases of about 85% respect to the unirradiated polymer. For helium this value is  $2 \times 10^{12}$  cm<sup>-2</sup> and for carbon  $4 \times 10^{11}$  cm<sup>-2</sup>. At these fluence values no sign of graphite was found by FTIR studies. Using a Monte Carlo simulation program we determined that the surface area affected by the track core of the incoming ions was less than 19 and 35% for helium and carbon respectively.

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## 1. Introduction

For many years, ultra high molecular weight polyethylene (UHMWPE) has been the choice as a bearing material for total joint replacement prostheses (TJR) owing to such excellent properties, as bio-compatibility, chemical stability and mechanical integrity [1]. However, UHMWPE wear and the resultant debris induce a series of adverse tissue reactions that cause osteolysis and premature failure.

Efforts have been made to enhance the wear resistance of UHMWPE. These include, among others, the performance improvement of this polymer by cross-linking methods that raise the UHMWPE wear resistance [2–4]. Generally cross-linking is

obtained by exposing the polymer to gamma or electron beam radiation.

Highly energetic gamma rays used for radiation treatments affect homogeneously the bulk material. On the other hand, the penetration depth of electron radiation strongly depends on its energy and the absorbing material density, e.g. a 10 MeV electron beam range is about 4 cm in this polymer [5]. It has been shown that cross-linking induced by high doses of gamma or e-beam radiation in UHMWPE dramatically reduce the abrasive and adhesive wear. However, these procedures alter the polymer structure. For instance, radiation treatments in bulk UHMWPE result in a reduction of several important mechanical properties including ductility, elastic modulus, fracture toughness and crack propagation resistance as mentioned in a recent review by Pruitt [6].

Ion beam irradiation is an effective surface modification technique, which uses heavy ions to alter the outermost surface of

\* Corresponding author. Tel.: +54 11 67727158; fax: +54 11 67727121.

E-mail address: [delgrosso@tandar.cnea.gov.ar](mailto:delgrosso@tandar.cnea.gov.ar) (M.F. del Grosso).

polymers without affecting their bulk properties [7]. This last argument has promoted the use of ion beams, provided by high voltage accelerators, to develop new applied techniques with the purpose to change the properties of UHMWPE in a controlled way.

When high energy ions interact with matter, during their passage through the material they lose their energy by excitation or ionization of atoms by inelastic collisions, known as Electronic Stopping, and the energy spent in this process is called electronic energy loss. Electronic Stopping is dominant in high energies where the displacement of atoms due to elastic collisions is insignificant. Such heavy ions, with energies so high that the electronic loss process dominates, are referred to as swift heavy ions (SHI).

The basic difference of materials' modification by low energy ions implantation (hundreds of keV) and swift heavy ion irradiation (greater than 1 MeV/amu) is that the low energy ions get embedded in the material and cause modification by their presence and due to the collision cascade produced by the impinging ions, whereas, in SHI irradiation, the modification of thin films or the near surface region of the bulk samples is due to the electronic excitation.

On their way through matter, SHI lose their energy to the electrons of the target leading to a continuous trail of excited and ionized target atoms along the ion path. The ejected electrons have a broad spectrum of kinetic energies and trigger a considerable number of further ionizations. Most of the primary processes occur close to the ion-trajectory in a cylindrical zone with a radius of a few nanometers, called "nuclear track" or "latent track". The energy deposited into such a small volume is extremely high and can reach several keV/nm<sup>3</sup>. Processes induced by the cascade of the secondary electrons are spread over a much larger radial distance from the track core. The maximum range depends on the projectile velocity and may reach up to a micrometer.

A number of articles have been published in the subject of ion implanted UHMWPE [8–13] that involves low energies. Some works study high stopping power effects in different polymers [14–17], but not in UHMWPE. To our knowledge little is known on physico-chemical and wear modification in UHMWPE with swift heavy ions, which is the aim of this work.

When an ion beam penetrates polyethylene it produces highly dense electronic excitations which induce complex physical and chemical processes. In UHMWPE, that has very long molecular chains, only a single incident ion is necessary to induce the bonding of two carbon atoms in two different long chains. As a consequence it leads to a longer macromolecule and a major change in physical, chemical and mechanical behavior.

In this work we analyzed the structural changes produced in UHMWPE by carbon and helium beams as a function of the irradiation fluence using Fourier transform infrared spectroscopy (FTIR) and performing wear resistance tests.

## 2. Experimental

### 2.1. Materials

Medical grade UHMWPE (GUR 1050) used in the present study was supplied by Poly Hi Solidur (Germany). The manu-

facturer quoted the material density as 0.932 g/cm<sup>3</sup>, yield strength 0.48 MPa and average molecular weight as 5.5 million g/mol. UHMWPE was stored in nitrogen to reduce any oxidative process.

We prepared two different samples to be used in different experiments:

Sample 1 Bulk UHMWPE was machined into parallelepipeds (10 × 15 × 6.3) mm. Prior to ion irradiation, one face was grounded with 600, 1000 and 1500 # grit SiC paper, then polished with diamond paste with particle sizes of 6, 3 and 1 μm following the process described by Riester et al. [18]. The polished pieces were cleaned using distilled water in an ultrasonic bath. This kind of sample was used in wear measurements.

Sample 2 Bulk pristine UHMWPE was microtomed with a Reichert-Jung Policut E Microtome in thin foils of approximately 10 μm thickness. We used a constant cutting velocity of 20 mm/s to reduce the heating of the samples. These samples were irradiated and finally FTIR measurements were performed.

### 2.2. Ion irradiation

Both samples, the bulk material and the thin films, were irradiated in a vacuum better than 10<sup>-7</sup> mbar with ion beams perpendicular to the specimen surface. The 12.5 MeV C and 6.77 MeV He beams were provided by the 3 MV Tandem of the Instituto de Física, UFRGS, Porto Alegre, Brazil. Irradiations were performed with fluences ranging between 2 × 10<sup>10</sup> and 5 × 10<sup>13</sup> cm<sup>-2</sup> for carbon and 10<sup>11</sup> and 10<sup>13</sup> cm<sup>-2</sup> for helium beam, respectively. The current density was less than 0.1 nA/cm<sup>2</sup>, which prevents sample heating.

### 2.3. Wear testing

Tribological behaviors of irradiated UHMWPE were measured using a ball-on-disk tester (CSEM Calotest). A 19 mm diameter AISI 316 steel ball was selected for counter wear. The ball rotational speed was 200 rev min<sup>-1</sup> and the load applied to the samples was 0.25 N, this value assures that plastic deformation is avoided. The balls were previously degreased with acetone. Wear tests were conducted during 20 min at room temperature, ambient humidity and without lubrication. The diameter of the wear crater was measured with a Philips, PSEM-500 Scanning Electron Microscope (SEM). UHMWPE volume loss was indirectly calculated using this measurement [19].

### 2.4. Infrared spectroscopy measurements

The acquisition of infrared spectra in the transmission mode was carried out at a resolution of 4 cm<sup>-1</sup> using a Nicolet Impact 410 spectrometer, equipped with a DTGS detector. Calculations for ion penetration were performed using the SRIM-2006 [20] program. The projected ion ranges are 48 and 20 μm for 6.77 MeV He and 12.5 MeV C, respectively. For FTIR measurements the

microtomed material was used. As mentioned above the sample thickness was kept around 10  $\mu\text{m}$  to allow the passage of the radiation through the whole material. To minimize aging the films were kept in vacuum before and after irradiation and until infrared spectroscopy analysis was performed.

The intensity measurements were properly normalized in order to take into account the variation of the sample thickness. We selected as a reference peak the methylene absorbance centered at 1367  $\text{cm}^{-1}$ , which depends on the amount of material intercepted by the infrared beam and is practically unaffected by irradiation [21].

### 3. Results

#### 3.1. Wear results

Heavy ion irradiation of UHMWPE induces changes in its mechanical, physical and chemical properties that depend, among other, on ion fluence. These modifications take place only on polymer surface and not in the bulk material. We used the CSEM tribometer to evaluate wear performance of irradiated UHMWPE. In Figs. 1 and 2 the experimental wear volume loss is plotted, taking an average of five measurements, as a function of ion fluence for He and C respectively. The irradiated modified zones are thicker ( $>20 \mu\text{m}$ ) than the crater depths ( $<5 \mu\text{m}$ ) produced by wear tests. As can be observed the Calowear tests showed that in general ion irradiation improves wear properties of this polymer. In fact, all the irradiated specimens present a lower loss volume than the pristine samples. Helium irradiated samples (Fig. 1) show a 85% wear reduction at  $2 \times 10^{12} \text{ cm}^{-2}$ .

On the other hand, for those samples irradiated with C beams (Fig. 2), we can see two regions where the loss volume decrease approximately an 85%, a sharp one at  $4 \times 10^{11}$  and a broad minimum between  $10^{13}$  and  $10^{14} \text{ cm}^{-2}$ .

In both figures the error bars represent statistical dispersion of the five measurements for each fluence.

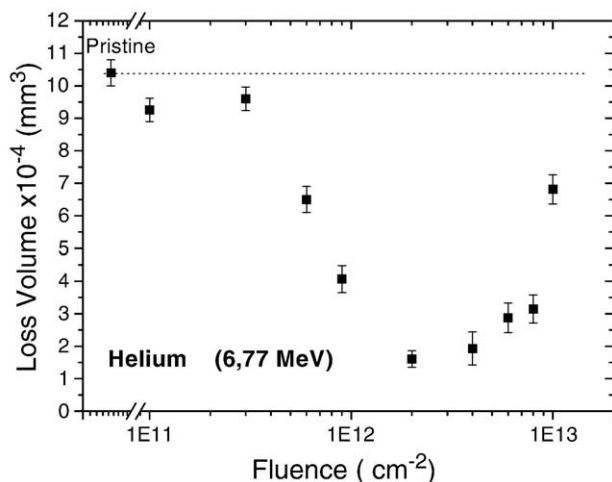


Fig. 1. Loss volume measurements of 6.77 MeV He irradiated UHMWPE versus ion fluence, compared with unirradiated sample.

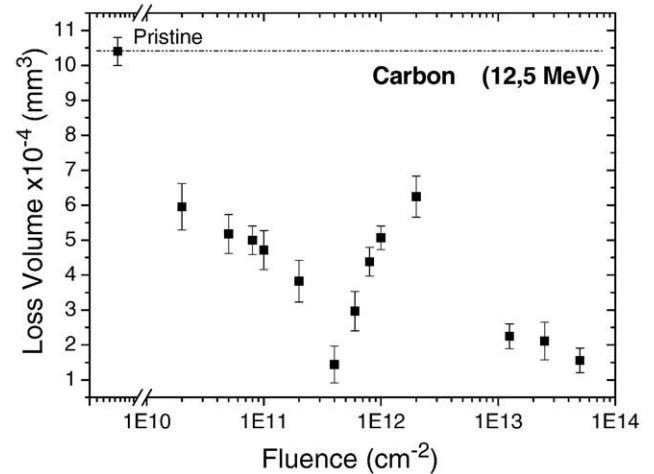


Fig. 2. Loss volume measurements of 12.5 MeV C irradiated UHMWPE versus ion fluence, compared to the pristine sample.

#### 3.2. Infrared study

There are several works that reported changes produced on polymers by irradiation. In particular, Balanzat et al. [22,23] investigated polyethylene modifications due to swift heavy ions and Tretinnikov et al. studied surface alterations of the UHMWPE irradiated in air with high energy pulsed electron beams [21]. Very few papers have been published on the subject of microtomy effects in polymers. Costa et al [24] found changes in crystallinity and orientation of the cutting samples. We have performed FTIR studies on unirradiated and irradiated films to compare them and to guarantee that all the modifications are due to the passage of the ion beam.

In this section we will use the FTIR technique to characterize some chemical effects induced on UHMWPE by swift heavy ion irradiation. Several FTIR spectra were measured for each fluence value and statistical dispersion is represented by error bars in the figures. In FTIR transmission measurements the normalization procedure is needed in order to account for the variation in the sample thickness. As mentioned above for UHMWPE the methylene absorbance,  $\text{CH}_2$  wagging peak, at 1363  $\text{cm}^{-1}$  was selected as a reference band, because it is strong, well resolved and remains practically unaffected by ion irradiation [21].

One of the main irradiation products on this polyethylene is the double bond  $\text{C}=\text{C}$  formation, which can occur in different chain positions. The creation of in-chain unsaturations are revealed by a peak at 965  $\text{cm}^{-1}$  that represents the trans-vinylene group ( $-\text{CH}=\text{CH}-$ ) while others such as the end chain, vinyl groups ( $-\text{CH}=\text{CH}_2$ ), present absorption at 910–994  $\text{cm}^{-1}$ .  $\text{C}=\text{C}$  stretching vibration at 1640  $\text{cm}^{-1}$  is usually a weak band, and rarely can be used to detect double bonds formation [21–23].

In our spectra the formation of trans-vinylene groups can be detected at 967  $\text{cm}^{-1}$ , its intensity grows with the fluence, but no signs of absorption at 910–994  $\text{cm}^{-1}$  (vinyl groups) can be found. On the other hand, the stretching band intensity at 1640  $\text{cm}^{-1}$  can be easily detected and quantified. This peak, as

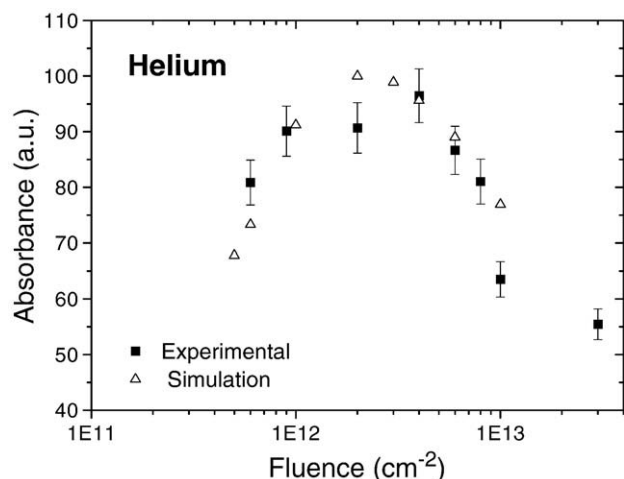


Fig. 3. C=C stretching band absorbance of irradiated UHMWPE with 6.77 MeV He beam with MC simulation results as a function of fluence. The absorbance maximum is at  $2 \times 10^{12} \text{ cm}^{-2}$ .

proposed by Tretinnikov [21], could come from  $-\text{CH}(\text{OOH})-\text{CH}=\text{CH}-\text{CH}_2-$ , unsaturation sites containing hydroperoxides in  $\alpha$ -position to trans-vinylene groups.

In Figs. 3 and 4, the absorbances of the C=C stretching peak (normalized to the methylene reference peak) are shown as a function of He and C irradiation fluence respectively. As can be observed the intensity increases passes through a maximum and then decreases for larger fluences.

In particular for carbon irradiation fluences greater than  $2 \times 10^{12} \text{ cm}^{-2}$  (Fig. 4) all the characteristic UHMWPE bands have disappeared.

Another characteristic band that come from the crystalline fraction is the  $\text{CH}_2$  rocking that presents a component at  $1470 \text{ cm}^{-1}$ . This band overlaps with the bending band at  $1460 \text{ cm}^{-1}$  that is originated from both crystalline and amorphous

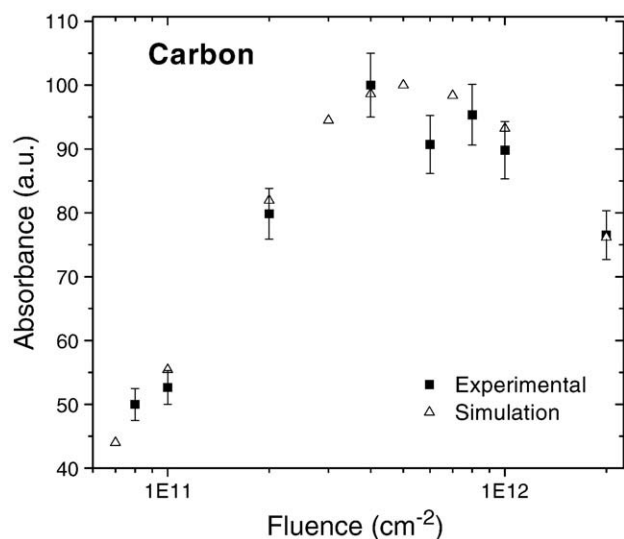


Fig. 4. C=C stretching band absorbance of irradiated UHMWPE with 12.5 MeV C beam with MC simulation results as a function of fluence. The absorbance maximum is at  $4 \times 10^{11} \text{ cm}^{-2}$ .

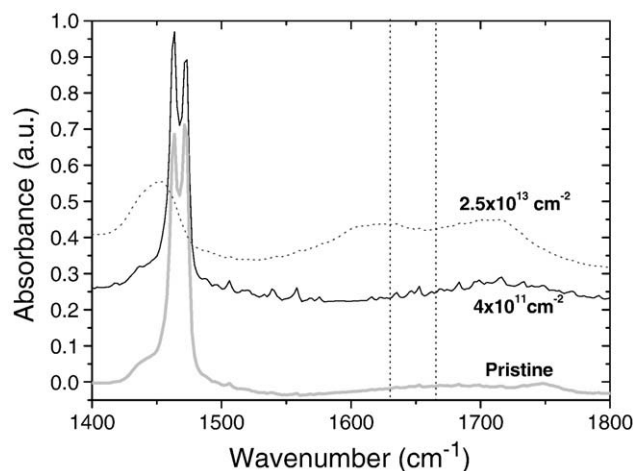


Fig. 5. FTIR analysis of pure UHMWPE and  $4 \times 10^{11} \text{ cm}^{-2}$  C irradiated. The spectra have been shifted vertically for clarity purposes.

phases, see Fig. 5. We detected changes in C–H peaks as the irradiation fluence increases: first a decrease in the  $1470$  and an increase in  $1460 \text{ cm}^{-1}$  bands. Then, for irradiation fluences greater than  $1 \times 10^{13} \text{ cm}^{-2}$ , the signal that represents the methylene content ( $1460 \text{ cm}^{-1}$ ) tends to disappear as can be seen in Fig. 5. This can be caused by an overall destruction of the polymer (breaking of C–H bonds) as described by Balanzat et al. [23]. The crystalline fraction presents also the bending vibration at  $730 \text{ cm}^{-1}$  that overlaps the rocking band vibration at  $720 \text{ cm}^{-1}$  (amorphous+crystalline), not shown here. These peaks display the same behavior as the ones described above.

It is well known that irradiation of UHMWPE with heavy ions induces de-hydrogenation and carbon enrichment, so the resulting surface could show a layer rich of “graphite-like” structure [25]. Related with this, we found at the higher fluences two broad peaks in  $1600\text{--}1700 \text{ cm}^{-1}$  region that are quite coincident with graphite C–C stretching peaks. In Fig. 5 dashed lines are placed at  $1632$  and  $1660 \text{ cm}^{-1}$ , where peaks related to graphite structures should appear. In addition we observed a change in color in the irradiated area for fluences larger than  $2 \times 10^{12} \text{ cm}^{-2}$ , shifting from white to yellow and dark brown, it could be caused by de-hydrogenization process. Furthermore, for such high fluences the samples become thin and fragile, making it hard to manipulate.

#### 4. Discussion

Ion beam irradiation is an effective surface modification technique. The energy deposited by the ions in the material is characteristic of the ion type and its energy. The kinetic energy transfer takes place in the latent track produced by the ions, where cross-linking and scission phenomena occur. New chemical bonds are also generated in the macromolecule, such as  $-\text{C}=\text{C}-$ , that result in an increase in the hardness of the material by restricting intramolecular motion.

When we studied double bond C=C formation and wear resistance as a function of the irradiation fluence, we found that the experimental results (see Table 1) from infrared spectroscopy

and wear tests presented a similar behavior. All of them reached a maximum (minimum) at almost the same fluence value  $\phi^*$ .

A similar effect was previously observed analyzing different irradiated polymers and measuring other parameters such as: the grafting process [26] or a particular chemical structure as mentioned before [27].

In that previous work we analyzed the results by means of a model developed by Mazzei et al. [28]. This model assumes that the effect of the ion bombardment on the polymer consists on a totally destructed core zone (nucleus) along the ion axis surrounded by a region in which a particular physico-chemical effect occurs (active zone). Then when two latent tracks intersect each other the deactivated zone of one latent track can eliminate the active zone of the other track. This mechanism can explain the reduction of the total measured physical quantities above certain ion fluence ( $\phi^*$ ). A Monte Carlo (MC) code, based on this simple model, that simulates the irradiation process from a geometrical point of view was developed [27] and the results (the total area affected by the ion tracks) are plotted together with experimental data in Figs. 3 and 4. As can be observed good agreement was found between the experimental and simulation values. From this analysis results that the radii for the nucleus and the active zone are for He 1 and 8 nm, and for C beam 2.5 and 15 nm, respectively.

We quantified the track overlapping with the Monte Carlo code mentioned above. This code can be used to examine the track distribution on the material surface and to calculate the overlap of both active and nucleus zones. Table 2 shows the sample area affected by the core zones. For both ions, we can see that less than 10% nucleus covering is obtained for fluences smaller or equal to  $\phi^*$ . For values higher than  $\phi^*$ , the curves behavior depends on the total area affected by the nucleus as can be seen in Figs. 3 and 4, even though it represents less than 40% at the highest fluences presented here. This means that most of the material has only been affected by the active zones of the tracks. Furthermore, at these fluences the FTIR spectra still present the polymer characteristic peaks, with no signs of graphitization.

We irradiated the carbon samples with fluences higher than  $1 \times 10^{13} \text{ cm}^{-2}$  and we observed that their color changed to dark brown and they became thin and fragile. Valenza et al. [25] modified UHMWPE by ion implantation at 300 keV. In that work they asserted that the observed partial de-hydrogenization is due to the sputtering process. This process leads to an increase in the amount of carbon concentration, resulting in a carbonized layer on the surface of the polymer. De-hydrogenization of the polymer is known to increase with ion fluence reaching a saturation value at  $2 \times 10^{13} \text{ cm}^{-2}$ .

Valenza [25] and Pawlak [29] stated in previous works that crystal structures are definitely destroyed when the fluence increases. For multiple overlapping tracks, the carbonization

Table 2

Percentage of surface area affected by nucleus tracks at several fluences

Ion	Fluences ( $\text{cm}^{-2}$ )	% nucleus covering
He	$5 \times 10^{11}$	2
He	$2 \times 10^{12}$	7
He	$6 \times 10^{12}$	19
C	$7 \times 10^{10}$	1.5
C	$4 \times 10^{11}$	8
C	$2 \times 10^{12}$	34

proceeds further, leading to an enrichment of sp<sup>2</sup> bonds (graphitization). They assert too that the great density differences between the original polymeric material and the carbonized irradiated areas due to the split-off of volatile components, finally produce excessive material tensions, which eventually might relax by crack formation.

Another of the consequences of radiation in UHMWPE is the generation of a three-dimensionally cross-linked network, which increases the rigidity of the chains changing the intermolecular bonds from Van der Waals to stronger covalent C–C bonds. For example, C–H bonds are broken, releasing H in gas form and free radicals leading to new C–C bonds. This is partly responsible for the reduction in wear of the irradiated material relative to the non-irradiated case. Cross-linking is not easily measured and it is hardly observed using FTIR techniques [22]. It is usually done by means of a swelling technique [30] which is not appropriate for our case, as it requires a minimum volume of  $3 \times 3 \times 3 \text{ mm}$  of uniformly irradiated material. This cannot be done in the present work where irradiation only affects the surface of the sample in a nonuniform or homogeneous way.

There are several advantages of using ion beams, instead of gamma or electron irradiation: a) the high energy deposition induces a highly cross-linked surface, b) the ion range is only several microns leaving most of the bulk material with its mechanical properties unaltered and c) the chemical change produced by the H out diffusion, that finally leads to the surface carbonization, is much more reduced using highly energetic ion beams and low fluences.

On the other hand, highly energetic ion beams should be provided by sophisticated accelerators, which represents a disadvantage. But taking into account that a single ion can link two large polyethylene macromolecules, a low current is needed and thus the irradiation time is in general reduced. For this reason, the surface treatment process could be practically feasible from an economical point of view.

## 5. Conclusions

In previous results [10,19,25,31] UHMWPE was modified using ion implantation that involves different energy ranges and also different physical processes than the ones presented here. In our work we have shown that ion beam irradiation at low fluences improves the mechanical resistance of the polymeric surface with minor changes in the material essence. We have determined that exists an optimum ion fluence value, that depends on the ion mass and energy, at which the wear resistance

Table 1  
Experimental results

Ion	Energy (MeV)	$\phi^*$ ( $\text{cm}^{-2}$ )	Wear reduction (%)
He	6.77	$2 \times 10^{12}$	85
C	12.5	$4 \times 10^{11}$	85

increases of about 85% respect to the unirradiated polymer. For helium this value is  $2 \times 10^{12} \text{ cm}^{-2}$  and for carbon  $4 \times 10^{11} \text{ cm}^{-2}$ . In addition, no signs of a graphite-like layer at the polymer surface is observed. From FTIR spectroscopy we found a maximum formation of double carbon bonds, at approximately the same fluence mentioned above for the increasing wear resistance in both helium and carbon irradiation. For fluences greater than  $1 \times 10^{13} \text{ cm}^{-2}$  for He and  $2 \times 10^{12} \text{ cm}^{-2}$  for carbon all the characteristic bands of UHMWPE have disappear and the surface color has turn to dark brown.

Concluding, to our knowledge the present work establishes the existence of an optimum ion fluence at which: 1) the surface of the irradiated material increases its wear resistance properties leaving the bulk material unaltered, 2) this improvement is correlated to other chemical changes induced by the high energy ion irradiation and 3) it strongly depends on the ion parameters. Finally, due to its practical relevance, the study of the wear properties as a function of ion fluence as well as other experimental parameters deserve an extensive and careful analysis in future investigations.

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