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Influence of irradiation doses on the mechanical and tribological properties of polyetheretherketone exposed to electron beam treatment

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ABSTRACT

This study investigated the effects of electron beam irradiation on the mechanical and tribological properties of polyetheretherketone (PEEK), with particular focus on modifications resulting from the application of the Litol-24 lubricant. Samples of pre-treated PEEK were irradiated at doses of 100, 200, 400, and 600 kGy using the ILU-10 linear accelerator. Comprehensive analyses were conducted, including thermogravimetric analysis (TGA) to assess thermal stability, X-ray diffraction (XRD) to observe structural changes, and the impact of irradiation on microhardness. Tribological performance was evaluated using the ball-on-disc method. Results indicate that irradiation decreases microhardness by approximately 19% and modifies the tribological behavior in a dose-dependent manner. TGA results showed subtle shifts in decomposition onset temperatures, with a reduction of about 10 °C post-irradiation, while XRD revealed a 12% decrease in crystallinity, affecting mechanical properties. Further investigations demonstrated that lubrication, particularly under high-load conditions, could enhance the operational characteristics of PEEK post-irradiation. The study underscores the critical role of lubricants in improving the wear resistance and durability of PEEK, making it suitable for high-stress applications in mechanical engineering and manufacturing sectors. The analysis highlighted the potential of integrating electron irradiation into existing material processing workflows to improve the properties of PEEK, thereby extending its utility across various industrial applications. This approach offers a promising avenue for optimizing the performance and longevity of PEEK components, particularly in the environments subject to extreme mechanical stresses.

Keywords: polyetheretherketone (PEEK), electron beam treatment, radiation dose, microhardness, friction, wear, lubricant.

INTRODUCTION

Polymers play a central role in the modern world, significantly influencing the development of many industrial sectors. Owing to their unique ability to be modified and adapted, polymeric materials are used in a wide range of areas—from medicine to the aerospace industry. Their appeal is due to high functional versatility, ease of production, and the ability to adapt mechanical and physicochemical properties to specific practical tasks. In particular, PEEK stands out among other polymers.

PEEK is a synthetic thermoplastic polymer that belongs to the family of PAEK, possessing a number of valuable properties for use in mechanical engineering. Its high strength and wear resistance make it an ideal material for manufacturing machine parts subjected to high loads. The excellent chemical resistance and X-ray transparency of PEEK allow its use in aggressive environments and facilitate diagnostics without disassembly. These qualities make PEEK a sought-after material in the field of high technology and mechanical engineering, for example, in the production of components for precision mechanisms or parts for special equipment. For instance, PEEK can replace metal, stainless steel, and titanium in engine casings, gaskets, bearings, clutch gears, seals, and other components; in addition, it can be used in transmissions, braking systems, and air conditioning systems [5–8].

Furthermore, the surface properties of PEEK can be enhanced to meet specific engineering requirements. The methods for modifying PEEK surface characteristics include the exposure to gamma rays [9–12], ion implantation [13–15], and ultraviolet radiation [16–18], which are well studied in the scientific literature. PEEK can also be processed by electron beam irradiation, which does not require the addition of fillers or additives. This method involves physically impacting the material with high-energy electron beams, allowing for the adjustment of intermolecular bonds and the surface topography of PEEK to improve its interaction with other materials in composite structures [19–20]. Electron irradiation can cause cross-linking of polymer chains, enhancing wear resistance and creep resistance of the material [21–23]. Electron beam processing can also affect the thermal stability of PEEK. The parameters for polymer processing can be adjusted by changing the dosage and energy of the electron beam [24–25]. To achieve the desired outcomes after irradiation, it is necessary to determine optimal parameters. Among the parameters of electron irradiation, the energy of the beam is important, as it determines the depth of electron penetration into the material, and the irradiation dose determines the total amount of energy transferred to the material. High beam energy allows electrons to penetrate deeper, which is important for processing thick materials, while a high dose of irradiation can cause more significant changes in the structure and properties of the material, such as increased hardness or changes in thermal and mechanical properties [26–28].

The irradiation dose, as a distinct and critical technological parameter, requires thorough investigation to understand its direct impact on material properties. This study focused on examining the effects of various electron beam irradiation doses on the mechanical and tribological properties of PEEK, with the aim of optimizing processing conditions and enhancing material performance. Researching the impact of electron irradiation on PEEK is a timely task, as the results not only expand understanding of the fundamental processes occurring in PEEK under the influence of electron irradiation but also propose practical solutions for improving its operational properties

MATERIALS AND METHODS

Material

The study utilized PEEK disks as the primary material. The disks were cut from a rod with a diameter of 30 mm and thickness of 3 mm. Before electron beam irradiation, the disks underwent preliminary grinding and polishing on the GP-1A machine using sandpaper of various grits, ranging from P600 to P2500. The initial average surface roughness (Ra) of the disks was 0.45 micrometers.

Electron beam treatment

The PEEK specimens underwent irradiation at the ILU-10 pulsed linear accelerator situated in the Nuclear Technology Park in Kurchatov, Kazakhstan (refer to Figure 1). The electron beam parameter settings were fine-tuned to obtain the desired

Figure 1. Pulsed linear accelerator ILU-10

radiation doses. Each pass achieved a dose of 10 kGy, and several passes were necessary to accumulate the necessary dose. Detailed specifications of the irradiation process are listed in Table 1.

Thermogravimetric analysis (TGA)

To assess the thermal stability of the polymer following irradiation, a thermogravimetric analysis (TGA) using the TGA-1250 instrument was performed. Samples of PEEK, each weighing approximately 10 mg, were positioned in a ceramic crucible under an inert atmosphere, with nitrogen serving as the purge gas. The samples were heated from ambient temperature up to 800 °C at a rate of 10 °C per minute. The mass loss corresponding to temperature changes was meticulously recorded during this heating sequence. The precision of the thermogram recordings was maintained within an error margin of ± 2 °C.

X-ray diffraction (XRD)

X-ray diffraction analysis was conducted using the X'Pert PRO diffractometer (Philips Corporation, Netherlands) to assess the effects of electron beam irradiation on the structural-phase properties of PEEK. The analysis measured crystallite sizes within the samples both before and after irradiation. The settings for the XRD included a diffraction angle range of 15° to 35°, a scan step of 0.02°, and an exposure duration of 1 second, employing Cu Kα radiation at parameters of 40 kV and 30 mA.

Microhardness

Microhardness measurements were conducted using the FISCHERSCOPE HM2000S system, in accordance with the DIN EN ISO 14577-1 standards. Displacements were accurately recorded to within 0.1 nm, and load precision was maintained at 4 mg. The margin of error for microhardness measurements did not exceed 2%. Each test involved subjecting the samples to a 1 N load for 20 seconds, with the indenter traveling at 2 m/s and performing a minimum of ten hardness assessments on different parts of the surface. The collected data were primarily processed using WIN-HCU® Version 7.1 software, developed exclusively by Helmut Fischer GmbH Institut für Elektronik und Messtechnik, based in Sindelfingen, Germany. The tool utilized for indentations was a Vickers diamond pyramid, featuring a 136° angle.

Tribology testing

In this research, Litol-24 was utilized as the lubricant due to its superior lubricating qualities, making it suitable for applications that demand dependable boundary lubrication. Litol-24's high adsorption capacity on metal surfaces significantly reduces wear and friction, even under high loads and low speeds. Comprised of mineral base oil, lithium soap-based thickeners, and various additives, Litol-24 improves anti-corrosion and antioxidation properties, displaying a dynamic viscosity of about 18.5 × 10-3 Pa·s at 40 °C. This ensures efficient flow and stability of the lubricant film under load, contributing to notable reductions in wear and enhanced equipment lifespan, particularly in industrial and automotive settings where robust metal surface protection is essential under harsh operating conditions. The tribological behavior of PEEK samples, before and after electron beam treatment, was evaluated using the 'ball-on-disk' testing method. An Anton Paar TRB3 tribometer was used for these tests, applying a 10 N load, setting a sliding speed of 0.1 m/s, and covering a test distance of 1000 meters, adhering to ASTM G99-959, DIN50324, and ISO 20808 standards. To reduce friction and wear, each sample was fully submerged in Litol-24. Repeated testing, a minimum of four times per sample, ensured statistical validity. The average coefficients of friction were noted along with their standard deviations. Figure 2 illustrates the experimental setup with a steel ball interacting with a rotating disk, where load is consistently applied through the ball to the disk. The rotational direction of the disk is marked by a red arrow. A 6 mm diameter 100Cr6 steel ball served as the counterbody. The contact

Table 1. Electron irradiation parameters for polymers

Sample	Beam energy, MeV	Beam current, I, mA	Dose per pass, kGy	Runs	Radiation dose, kGy
PEEK-1	2.7	6.84	10	10	100
PEEK-2	2.7	6.84	10	30	300
PEEK-3	2.7	6.84	10	60	600

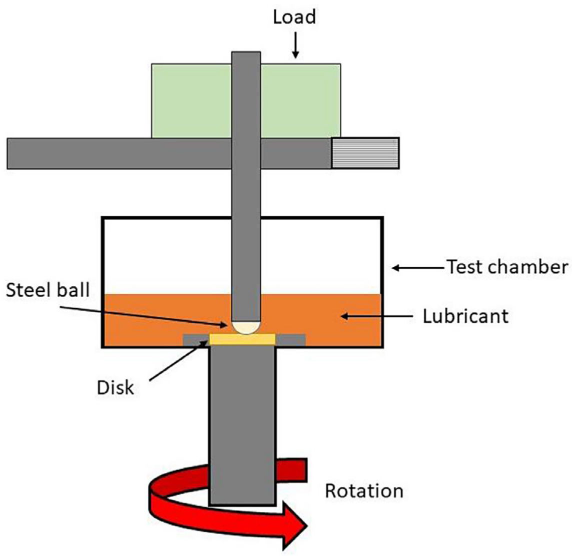

Figure 2. Schematic of the test rig for the ball-on-disk method using lubrication

surface area for the PEEK samples was 706.5 mm². Wear volume was evaluated using a Taylor Hobson Surtronic S-128 profilometer, facilitating automated data processing. Friction track profiles were measured five times at varied sample points post-testing to calculate average values. The wear volume from sliding was calculated using the formula 1 provided.

$$I = \frac{2\pi r S}{d \cdot P} = \frac{V}{d \cdot P} \quad (1)$$

where: r – the radius of the counterbody, S – the cross-sectional area of the wear track, V – the volume of material removed, d – the total testing distance, P – the applied load [29].

RESULTS AND DISCUSSION

Evaluation of thermogravimetric properties

Figure 3 presents the TGA curves, illustrating the weight loss of PEEK in a nitrogen atmosphere. The results show that the decomposition temperature of PEEK remained largely unchanged before and after electron irradiation. Thermal decomposition begins at approximately 520 °C, with the peak temperature for maximum weight loss around 650 °C for both the initial and irradiated samples (PEEK-1 and PEEK-2). Notably, PEEK-2 exhibits less mass loss at an irradiation dose of 300 kGy compared to other irradiated samples, indicating improved thermal stability. This improvement is likely due to molecular cross-linking, which restricts the mobility of macromolecules, thereby reducing their degradation. Beyond 700 °C, nearly complete combustion of the organic components occurs, which is typical of the pyrolytic processes in PEEK. Variations in the decomposition curves among the samples may result from differing levels of cross-linking and structural organization, influenced by the irradiation doses [30–34].

X-ray diffraction examination

Figure 4 illustrates the X-ray diffraction (XRD) patterns for both the initial and irradiated PEEK samples, examined over a 2θ range from 10° to 35°. The primary diffraction peaks, corresponding to the crystalline planes (110), (111), (200), and (211) of PEEK, appear at 2θ angles around 18.7°,

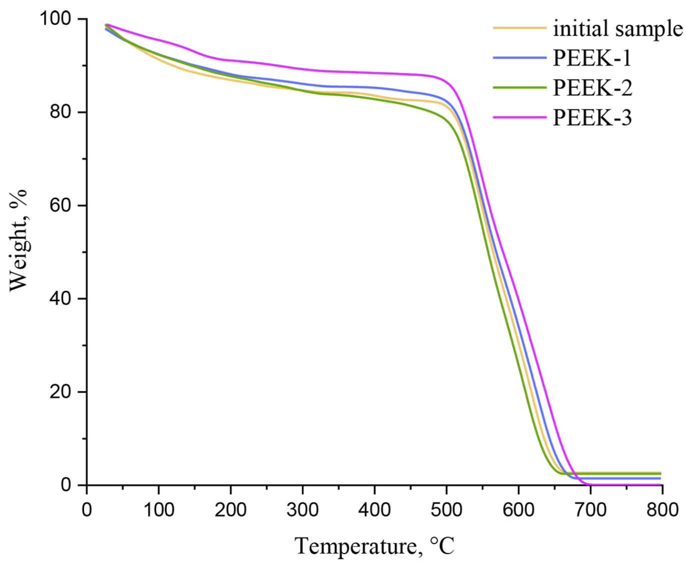

Figure 3. TGA curves of PEEK samples before and after irradiation

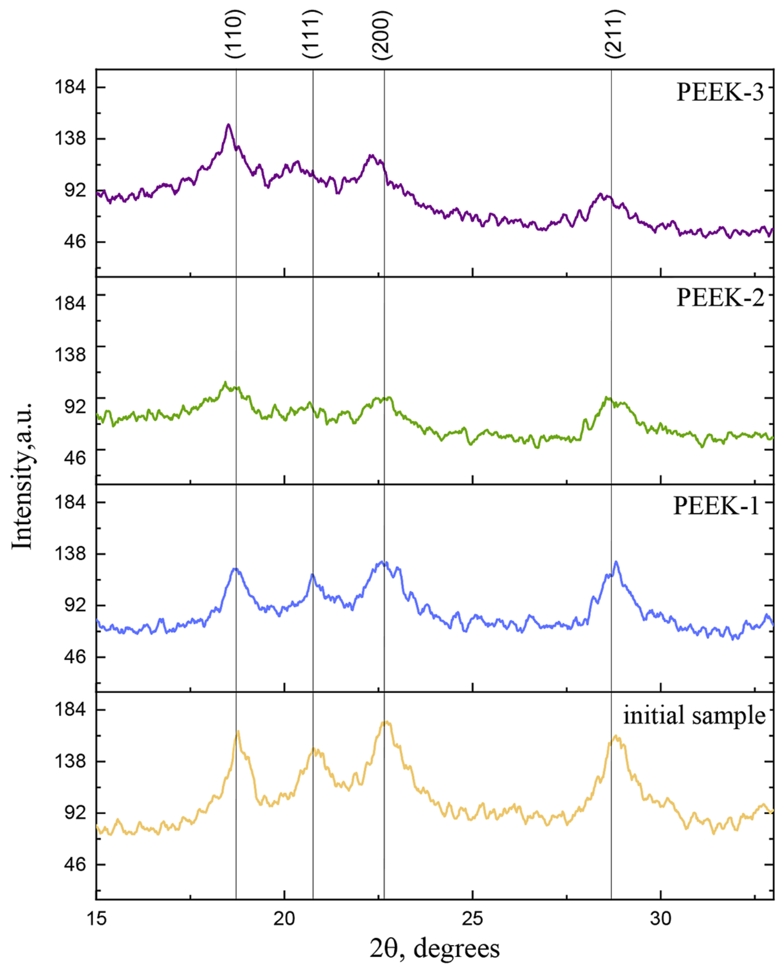

Figure 4. X-ray diffraction pattern of samples before and after irradiation with an electron beam

20.6°, 22.7°, and 28.7° [35]. The XRD profile for the PEEK-1 sample remains mostly consistent with the initial sample in terms of peak intensity and form. However, PEEK-2 and PEEK-3 display notable changes in peak heights compared to the baseline, indicating alterations in the crystalline structure and the formation of new intramolecular bonds. The presence of broad crystallization peaks suggests that the samples are semi-crystalline. The consistency of diffraction patterns across different irradiation doses implies that electron beam treatment influenced the crystalline structure of PEEK. Changes in peak height and breadth at varying irradiation doses may reflect shifts in the ratio of amorphous to crystalline phases. Literature suggests that crystallinity significantly impacts the mechanical and tribological properties of polymers [36]. Typically, exposure to high-intensity radiation induces a complex process where the polymer interacts with energetic electrons. The energy exceeds the bonding strength of electrons with atoms, leading to bond breakages and the formation of radicals. This process can result in cross-linking, chain scission, and various disproportionate reactions, alongside the production of gaseous molecules.

Impact of Irradiation on microhardness

The investigation confirmed that electron irradiation significantly influences the mechanical properties of PEEK, particularly impacting its hardness. Observations from Figure 5 demonstrate that microhardness adjusts proportionally to the irradiation dose administered. The study corroborates that alterations in the crystalline structure of the material substantially affect this mechanical property. The initial PEEK sample exhibited the highest hardness, reflecting its intact crystalline structure. Following irradiation, samples PEEK-1, PEEK-2, and PEEK-3 showed a reduction in hardness by approximately 19% compared to the initial specimen, with values around 155 MPa, 150 MPa, and 145 MPa, respectively. This consistent decrease in microhardness highlights the dose-dependent nature of the impact

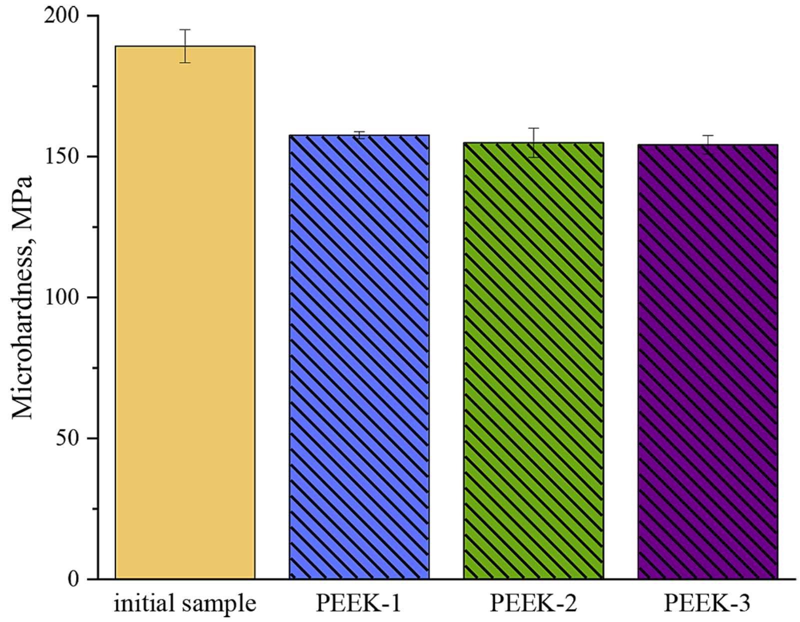

Figure 5. Changes in micro-hardness of PEEK samples before and after irradiation

of irradiation on the material. This represents an approximate 19% decrease in microhardness compared to the initial sample. The reduction in hardness can be attributed to changes in the molecular structure of the material under irradiation. These modifications are primarily due to the effects of irradiation on the microstructure of PEEK, particularly impacting its aromatic rings and ketone groups, which are characterized by bonds with very high activation energies and thus are more resistant to radiation-induced damage. Within the repeating units of the main chain, there are two carbon-carbon (C-C) and four carbon-oxygen (C-O) bonds with activation energies of 347 kJ/mol and 360 kJ/mol, respectively. These bonds are the most likely sites for radical generation, leading to the formation of new active chain ends that exhibit increased mobility, potentially recombining or establishing cross-links with neighboring molecules. Moreover, the scission of polymer chains and the formation of cross-links are primary factors affecting the mechanical properties. Irradiation prompts six of the twelve carbon-hydrogen (C-H) bonds to initiate the formation of new intramolecular bonds within the main chain across aromatic rings, while the other six bonds promote intermolecular cross-linking with adjacent molecules. This formation of intramolecular bonds, combined with some degree of cross-linking, reduces the flexibility of the main chain. The occurrence of chain scission creates new chain ends, further contributing to the decrease in microhardness. At higher doses of irradiation, cross-linking becomes the dominant process, significantly reducing mechanical properties of the polymer [37-38]. Despite the reduction in microhardness, the irradiated PEEK samples may still be suitable for industrial applications where flexibility and wear resistance are more critical than hardness. The ability to tune the mechanical properties of PEEK through controlled irradiation opens up potential for its use in environments where tailored performance is required.

Assessment of tribological behavior

Figure 6 illustrates the variation in the coefficient of friction (COF) for PEEK samples pre- and post- electron irradiation with the application of Litol-24 lubricant. The initial PEEK sample exhibits a friction coefficient of 0.108 ± 0.001, reflecting its inherent tribological characteristics. The PEEK-3 sample, which received the highest irradiation dose of 600 kGy, maintains a comparable friction coefficient, suggesting no notable enhancement or potentially even a degradation in its tribological performance following treatment. Conversely, the PEEK-1 sample, exposed to a lower irradiation dose, records the highest friction coefficient among the samples at 0.147 ± 0.005, implying subtle alterations in the properties of polymer surface that do not markedly enhance tribological outcomes. The PEEK-2 sample, irradiated at 300 kGy, registers the lowest friction coefficient at 0.097 ± 0.004, signifying beneficial structural modifications that aid in reducing friction, potentially due to optimal

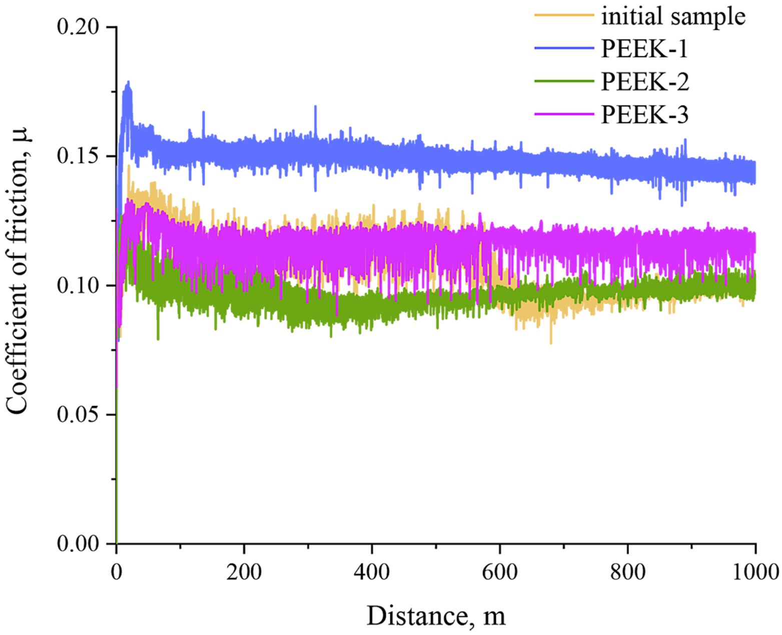

Figure 6. Friction coefficient of PEEK samples before and after irradiation

alterations in surface chemistry or structure following irradiation, enhancing its interaction with the Litol-24 lubricant. Although the PEEK-3 sample demonstrates improved tribological properties, its COF remains higher than that of PEEK-2, indicating that the irradiation dose may have surpassed the optimal level for achieving the most favorable properties. The employment of Litol-24 in these experiments highlights the critical role of lubricant selection in evaluating the tribological properties of polymers, particularly after modification through electron irradiation. Further investigations [39–40] into the effects of solid lubricants and the performance of PEEK in marine environments under frictional conditions have shown that lubrication markedly influences the friction coefficient and can extend the usability of irradiated PEEK in industrial applications by enhancing wear resistance and diminishing friction.

Figure 7 presents the variations in the wear rates of PEEK samples before and after exposure to electron irradiation, using Litol-24 as a lubricant.

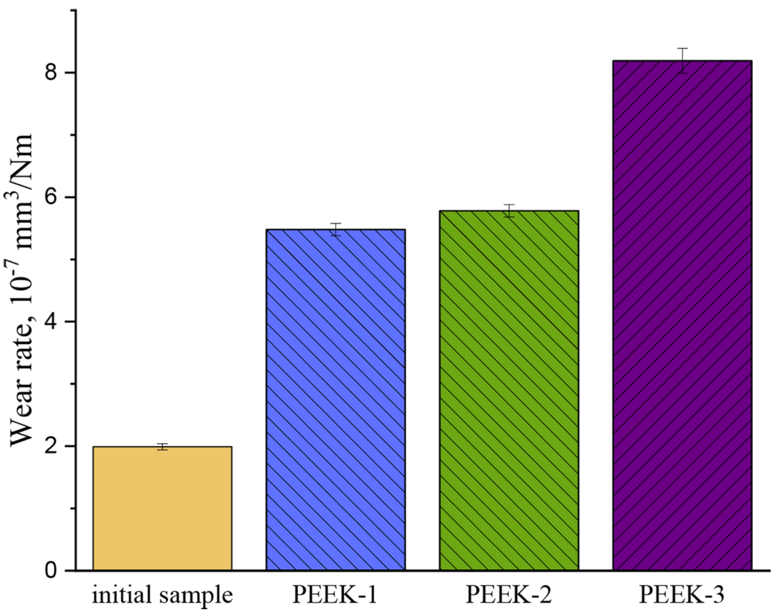

Figure 7. Wear rate of PEEK samples before and after irradiation

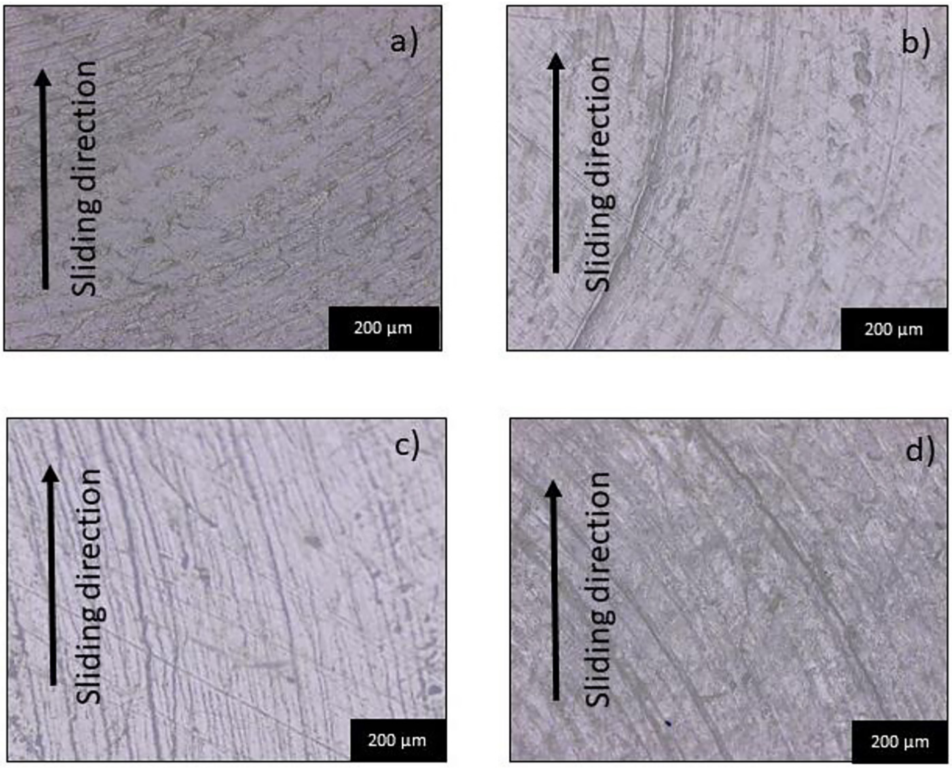

Figure 8. Wear tracks of initial and irradiated PEEK samples

The initial PEEK sample displays the lowest wear rate, underscoring its inherent high wear resistance. This correlates with its superior microhardness, highlighting its capacity to withstand mechanical stress without significant alteration.

As the irradiation dose increases, the wear rates for the PEEK-1, PEEK-2, and PEEK-3 samples show a progressive rise. The PEEK-1 sample, irradiated at a dose of 100 kGy, exhibits a slight increase in wear rate, potentially due to surface and structural changes that affect its interaction with the lubricant. For PEEK-2 and PEEK-3, irradiated at 300 kGy and 600 kGy, respectively, more pronounced increases in wear rates are observed. This suggests that significant structural modifications compromise the mechanical properties of the material, leading to reduced microhardness and overall wear resistance.

Figure 8 shows the wear track images of PEEK samples (a) initial sample, (b) PEEK-1, (c) PEEK-2, and (d) PEEK-3. The sliding direction is indicated by arrows. For the initial sample, the wear track exhibits minimal scratches and limited damage, reflecting its superior wear resistance. Conversely, for PEEK-1, PEEK-2, and PEEK-3, the sliding marks become more pronounced, with increasing groove depth and width as the irradiation dose increases. These visible changes in the wear tracks correspond with the escalating wear rates shown in Figure 7, further emphasizing the impact of irradiation on the tribological performance of the material.

The microstructural damage, evidenced by the wear track patterns, highlights the gradual breakdown of PEEK integrity under irradiation. The combination of higher wear rates and more severe sliding marks suggests that irradiation not only reduces microhardness but also accelerates surface degradation [11, 41–42]. Although the lubricant provides some mitigation against wear, it cannot fully counterbalance the adverse effects of radiation-induced structural alterations.

CONCLUSIONS

Electron beam treatment significantly impacted the physical and mechanical properties of PEEK, notably reducing its microhardness and wear resistance. The study of irradiation with different doses aimed to identify the consequences of structural changes and their impact on tribological characteristics. The TGA curves indicate that the decomposition temperature of PEEK remains relatively stable before and after electron irradiation. Among the irradiated samples, PEEK-2, subjected to a dose of 300 kGy, shows a lower degree of mass loss, indicating improved thermal stability. The observed differences in the decomposition curves can be explained by varying degrees of cross-linking and structural organization resulting from different irradiation doses. X-ray diffraction studies further clarified the changes in crystallinity, which directly correlate with microhardness. These results suggest that while irradiation improves certain aspects of the structural integrity of the material, it also leads to a certain brittleness.

Tribological tests revealed significant changes in the coefficient of friction and wear volume of the electron beam-treated samples. The friction coefficient measurements show that irradiation can both positively and negatively affect the tribological properties of PEEK depending on the applied dose. The PEEK-2 sample, irradiated at 300 kGy, exhibited the lowest friction coefficient, suggesting that this dose may optimize the surface properties of the polymer for reduced friction when used with Litol-24 lubricant. Conversely, the PEEK-1 and PEEK-3 samples, irradiated at 100 kGy and 600 kGy, respectively, did not show significant improvements, and in the case of PEEK-1, even an increase in friction was observed. This suggests that higher or lower irradiation doses may not be as effective in enhancing tribological performance. Wear rate analysis further corroborates these findings, with initial PEEK showing the lowest wear rate, indicative of its inherent high wear resistance. As the irradiation dose increases, a corresponding rise in wear rates is observed, particularly in the PEEK-2 and PEEK-3 samples. This suggests that while some irradiation can improve frictional properties, excessive irradiation may lead to detrimental structural changes, reducing the microhardness and overall wear resistance of the material.

Overall, the study highlight the critical balance required in selecting the appropriate irradiation dose to enhance the tribological properties of PEEK. Excessive irradiation can undermine the structural integrity of the polymer, leading to increased wear and decreased microhardness. The use of lubricants such as Litol-24 plays an important role in mitigating some of these effects, but it cannot fully counteract the negative consequences of high-dose irradiation. This research emphasizes the importance of carefully optimizing irradiation parameters to preserve or enhance the mechanical and tribological properties of PEEK for industrial applications.

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